AMERICAN COLLEGE OF PROSTHODONTISTS

Microtensile Bond Strength of Porcelain Laminate Veneers Bonded to Fluorosed Teeth

Priyadarshanee M. Ratnaweera, BDS, PhD, Nao Fukagawa, DDS, PhD, Yuji Tsubota, DDS, PhD, & Shunji Fukushima, DDS, PhD

Department of Fixed Prosthodontics, Tsurumi University School of Dental Medicine, Yokohama, Japan

Keywords
Microtensile bond strength; porcelain laminate veneers; fluorosed teeth.

Correspondence
Priyadarshanee Ratnaweera, 3/17A, Wheturangi Rd. Greenlane, Auckland 1005, New Zealand. E-mail: manorika@yahoo.com.sg, manorika@xtra.co.nz

This research was supported by a grant from the Japan Society for the Promotion of Science (No: 17-05244).

Accepted February 5, 2008

doi: 10.1111/j.1532-849X.2008.00403.x

Abstract

Purpose: The aim of this study was to evaluate the microtensile bond strengths (MTBSs) of porcelain laminate veneers bonded to normal and fluorosed teeth.
Materials and Method: Forty human incisors, including 20 normal and 20 moderately fluorosed teeth [Thylstrup Fejerskov Index (TFI) = 4 to 6], were collected. The labial surfaces of the teeth were ground up to 1 mm and polished with #600 silicon carbide abrasive paper. The surfaces were bonded to 1-mm-thick ceramic slices (5×5 mm^2) previously made (VitaVM7) using one of two ceramic cement systems (RelyX or Clapearl) following the manufacturers' instructions. A resin composite was added on top of the ceramic slices and built up to 5-mm thickness to serve as grips. The specimens were stored in water (37°C) for 24 hours, and beams of adhesive interface with a surface area of $\sim$1.25 mm^2 were obtained. Then the beams were subjected to MTBS tests at a crosshead speed of 1.0 mm/min. The data were analyzed with two-way ANOVA.
Results: The results of the MTBS test (MPa) were 20.55 ± 5.83 (RelyX/fluorosed), 20.16 ± 4.61 (RelyX/normal), 18.74 ± 2.88 (Clapearl/fluorosed), and 21.06 ± 4.99 (Clapearl/normal). There were no significant differences in the MTBSs among the four groups ($p > 0.05$).
Conclusions: The MTBSs of ceramic cement systems used were not influenced by the moderately fluorosed teeth.

Fluorosed enamel is featured histopathologically as a subsurface hypomineralized lesion covered by a well-mineralized outer enamel surface caused by continuous ingestion of excess fluoride during development of teeth.[1] This results in various pathological changes in the structure of teeth, characterized as mild fluorosis, which has an appearance of white flecking or spotting of the enamel or white opaque areas. Moderate to severe fluorosis has a brownish appearance, which can be combined with some white opaque spots and/or pitting of the enamel surface. Very severe fluorosis has very pitted and discolored enamel, giving rise to a corroded appearance. In cases of moderate and severe fluorosis the enamel may be more susceptible to wear and fracture.[2] Therefore, this condition gives rise to cosmetic problems of teeth in affected individuals. From Sri Lanka's population of about 20 million, 2 million people are at risk of getting dental fluorosis. Only 30% of the total population has clean piped water with controlled mineral content. Concentration of fluoride in well water exceeds 1.5 mg/l, sometimes as high as 10 mg/l in some parts of the country. Therefore, dental fluorosis is a massive social and oral health problem.[3]

Improvements in the properties of ceramics and porcelain bonding systems are responsible for porcelain laminate veneers becoming a standard treatment in restorative dentistry.[4] Current ceramic bonding systems are based on mechanochemical bonding between the luting material and ceramic restorations.[5] Fluorosed teeth are usually restored with tooth-colored restorations, such as composite resins or ceramic veneers. The best option to mask the discoloration of moderate to severe fluorosed teeth is to use porcelain laminate veneers.[6] The durability and clinical success of porcelain laminate veneers has been studied extensively, reporting a success rate of more than 95% with an average durability of over 5 years;[7] however, there have been no studies done on bond strengths of porcelain laminate veneers bonded to fluorosed enamel. Therefore, the aim of this study was to evaluate the microtensile bond strengths (MTBSs) of porcelain laminate veneers bonded to normal and fluorosed enamel and examine the bonding interfaces of the restorations by means of scanning electron microscopy (SEM). The hypothesis tested was that the MTBSs of porcelain laminate veneers differ according to the type of enamel.

Table 1 Materials used

Product	Components
Porcelain	
VitaVM7 (VITA ZahnFabrik, H. Rauter GmbH & Co. KG, Bad Sackingen, Gemany)	Feldspar, SiO_2, water
Veneer cements	
Clapearl (Kuraray Medical Co., Tokyo, Japan)	Bis-GMA, TEGDMA, methacrylate monomer, quartz powder, micro filler, optical polymerization catalyst, colorant
RelyX veneer cement (3M ESPE, Minneapolis, MN)	Bis-GMA, TEGDMA, Zirconia/silica and fumed silica fillers, dimethacrylate polymer, pigments, photoinitiator
Adhesive agents	
Photo Bond Catalyst (Kuraray Medical Co., Tokyo, Japan)	Bis-GMA, MDP, HEMA, hydrophobic dimethacrylate, benzoyl peroxide, dl-camphorquinone
Photo Bond Universal (Kuraray Medical Co., Tokyo, Japan)	*n,n'*-Diethanol *p*-toluidine, sodium benzen sulfinate, ethyl alcohol
Single Bond (3M ESPE, Minneapolis, MN)	Ethanol, HEMA, Bis-GMA, other dimethacrylate resins, methacrulate-modified polycarboxylic acid copolymer, water, photoinitiator
Silane-coupling agents	
Clearfil porcelain bond activator (Kuraray Medical Co., Tokyo, Japan)	Bisphenol-A-polyethoxy dimethacrylate, 3-methacryloyloxypropyl trimethoxy silane
Ceramic Primer (3M ESPE, Minneapolis, MN)	Prehydrated silane-coupling agent, alcohol, water
Etchants	
K-etchant (Kuraray Medical Co., Tokyo, Japan)	Phosphoric acid, colloidal silica, water
Scotchbond (3M ESPE, Minneapolis, MN)	Phosphoric acid gel (35% w/w)
Resin composite	
Clearfil ST (Kuraray Medical Co., Tokyo, Japan)	Silanated barium glass, silanated colloidal silica, Bis-GMA, benzoyl peroxide, dl-camphorquinone, hydrophobic aromatic dimethacrylate

MDP = 10-methacryloloxydecyl dihydrogen phosphate; Bis-GMA = Bisphenol A-diglycidylmethacrylate; HEMA = 2-hydroxyethyl methacrylate; TEGDMA = triethyleneglycol dimethacrylate; SiO_2 = Silicon dioxide.

Materials and methods

The materials used in this study are shown in Table 1. Forty human incisors, including 20 normal and 20 moderately fluorosed teeth [Thylstrup Fejerskov Index[8] (TFI) = 4 to 6] from patients living in areas endemic for fluorosis in Sri Lanka, were collected. These teeth were cleaned and stored in distilled water in a refrigerator at 4°C. All the teeth belonged to patients aged between 20 and 40 years. Informed consent was obtained from the patients whose teeth were used in this study.

Tooth preparation

The labial surfaces of the teeth were prepared to accommodate veneers of equal thickness. They were ground up to 1 mm and polished with #600 silicon carbide abrasive paper under water coolant and cleaned ultrasonically in distilled water for 5 minutes.

Ceramic veneer fabrication

The veneers were 1-mm thick, 5-mm high, and 5-mm wide (VitaVM7). These ceramic slices were made by a technician with the layering technique, following manufacturer's instructions.

Bonding ceramic veneers

The ceramic veneers were treated according to manufacturer's instructions, and the tooth surfaces were bonded using one of two resin cement systems for veneer following the manufacturer's instructions to the ceramic slices previously made. Resin cement systems used were RelyX veneer cement system and Clapearl. After each individual treatment of the tooth surface and ceramic, the ceramic veneers were placed on the prepared teeth with light finger pressure, and excess cement was removed with the brush. Photo-polymerization was performed with the light-polymerizing unit (Hyperlightel DPC-120, Morita, Tokyo, Japan) at 350 mW/cm^2 for 40 seconds for incisal, mesial, and distal surfaces.

To ensure that subsequent beams would have grips for attachment to the testing apparatus, a resin composite (DC Core Automix, Kuraray Medical Co.) was added onto the ceramic slices and built up to 5 mm thickness.

Specimen preparation

After cementation, the specimens were stored in distilled water for 24 hours in an incubator at 37°C. Each tooth was embedded in an acrylic resin block (Unifast II, GC, Tokyo, Japan) so the root of the tooth was inside the resin block and its coronal part was visible from the cementoenamel junction. These acrylic blocks were trimmed until they fitted the mounting device of the cutting machine, without manipulating the specimen teeth jutting out of the blocks, and prepared for sectioning in the slow-speed diamond saw sectioning machine with a diamond-rim blade (Isomet, Buehler, Lake Bluff, IL). Each

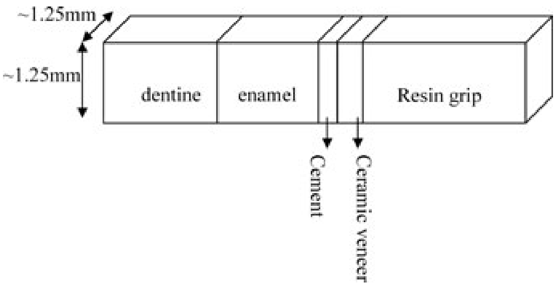

Figure 1 Schematic illustration of a prepared specimen.

Table 2 Microtensile bond strengths to enamel (MPa ± SD)

	Normal	Fluorosed
RelyX/VITA VM 7	20.16 ± 4.61	20.55 ± 5.83
Clapearl/VITA VM 7	21.06 ± 4.99	18.74 ± 2.88

$n = 30$ for each group. No statistically significant difference in the tensile bond strengths between the four groups ($p > 0.05$).

tooth was sectioned perpendicular to the bonded interface to obtain ∼1.25-mm thick slices, and each slice was further sectioned into beams to obtain an adhesive interface with a surface area of ∼1.25 mm^2 (Fig 1). Each beam was measured with digital calipers (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan) to ensure their thicknesses were accurate to within 0.05 mm.

Microtensile bond strength testing

After sectioning, each specimen was tested individually by one operator. Before testing, all specimens were checked under an optical microscope (magnification ×30) for bonding defects. Specimens showing formation of apparent interfacial gaps, air bubble formations, or any other defects were excluded from the study and replaced with new specimens. Cyanoacrylic adhesive (Zapit, Dental Ventures of America, Corona, CA) was used to attach the microtensile specimens to opposing arms of the microtensile testing device (Shimadzu, Tokyo, Japan). The specimen was fractured under tension at a crosshead speed of 1 mm/min, and the maximum load at fracture (N) was recorded.

Failure modes

Following bond strength testing, all fractured enamel/cement/porcelain surfaces were examined under a digital microscope (VHX 200, Keyence, Osaka, Japan) at original magnification ×40 to identify the failure mode. The failure modes were categorized into one of the following six types: (1) cohesive failure in the enamel; (2) cohesive failure in the bonding resin; (3) cohesive failure in the porcelain; (4) adhesive failure between the bonding resin and enamel; (5) adhesive failure between the bonding resin and porcelain at the bonded interface; and (6) mixed failure.

Statistical analysis

The ultimate stresses (MPa) of the porcelain/enamel bonds were calculated, and the results were analyzed with two-way ANOVA ($p < 0.05$) (SPSS/PC, Version 13.0; SPSS, Chicago, IL).

SEM observations

In addition, three specimens of each group were prepared for SEM analysis in the same manner as the MTBS test. To observe the interfaces between enamel and the adhesive resin, four bonded enamel/resin/porcelain specimens were cross-sectioned at the interfaces after storing for 24 hours at 37°C. The specimens were polished with polishing sheets (Imperial, Sumitomo 3M, Tokyo, Japan) in a descending sequence up to 3-μm roughness. They were then etched with 0.1 N HCl for 30 seconds and washed with distilled water. The specimens were dried in an incubator for 24 hours. Finally, the surfaces were sputter-coated with gold (SC-701AT, Quick Auto Coater, Sanyu Electron Inc., Tokyo, Japan) and observed under a scanning electron microscope (JSM 5600LV, JEOL, Tokyo, Japan).

Results

Micro-shear bond strengths and failure modes

Mean MTBS values and standard deviations in MPa are shown in Table 2. Two-way ANOVA revealed no statistically significant interaction between the two types of enamel and two resin cement systems used ($F = 2.481$, $p = 0.118$). Modes of failure following the micro-shear bond strengths are summarized in Table 3. Chi-square test showed no significant association between modes of failure with the two resin cement systems (asymmetric significance, two-sided: 0.935). However, adhesive failure between the bonding resin and enamel was the most prevalent type of failure for both types of resin cement systems in fluorosed enamel specimens. Specimens of normal teeth bonded to both types of cement systems reveal cohesive failure in the porcelain as the most prevalent type.

SEM observations

SEM photomicrographs of the adhesive interface between enamel, resin cement systems, and the porcelain are shown in Figures 2 and 3. There is no discontinuity in the enamel–resin

Table 3 Mode of failure after microtensile bond testing

			Failure mode					
Adhesive	Enamel	Number of specimens	1	2	3	4	5	6
RelyX	Normal	30	0	0	11	9	6	4
	Fluorosed	30	0	0	9	11	5	5
Clapearl	Normal	30	0	1	15	7	4	3
	Fluorosed	30	0	0	9	15	5	1

Categories: 1, cohesive failure in the enamel; 2, cohesive failure in the bonding resin; 3, cohesive failure in the porcelain; 4, adhesive failure between the bonding resin and enamel; 5, adhesive failure between the bonding resin and porcelain at the bonded interface; 6, mixed failure.

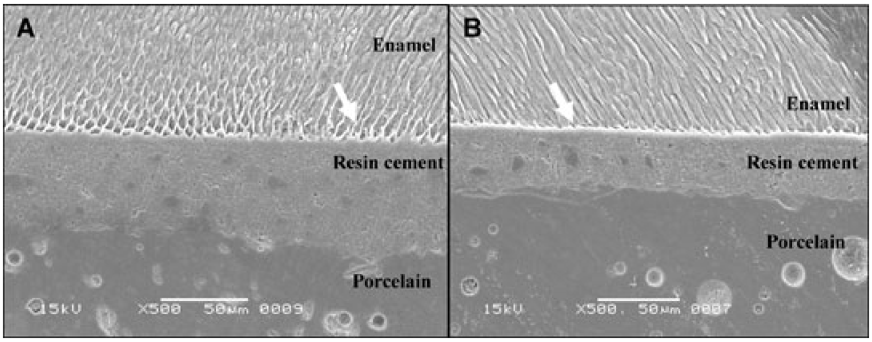

Figure 2 SEM images of adhesive interface of porcelain veneer bonded to fluorosed (A) and normal (B) enamel with Clapearl showing about 7 μm of resin tag-like penetrations in the enamel (arrows).

interface in either cement system, but the porcelain–resin bonding area sometimes exhibited a disrupted interface. Interface studies showed about 7-μm resin tags in the hybrid layer in enamel. No clear differences were observed in the hybrid layer of the two cement systems.

Discussion

Enamel fluorosis is characterized by a hypermineralized, acid-resistant superficial layer and porous enamel in areas of subsurface hypomineralization.[9] The prevalence of fluorosis in nonendemic areas is increasing in many parts of the world. This has been attributed to high background exposure to fluorides from various sources such as food, soft drinks, dentifrices, and fluoride supplements.[10,11] In endemic areas of Sri Lanka with high level of fluoride in water (more than 1 ppm), prevalence rates of fluorosis ranging from 29% to 57% have been reported.[12] For the classification of fluorosis, the modified TFI is very useful, as it is based on clinical changes in fluorosed teeth. The index is consistent with the histopathological changes in enamel. Since reproducibility is high, it is very useful in the studies of fluorosed teeth.[13] Moderately fluorosed teeth classified according to this index were used in this study.[8] Moderately fluorosed teeth consist of a marked opacity or chalky white appearance on the affected tooth surface and sometimes pits of less than 2 to 3 mm in diameter on the smooth surfaces or occlusal surfaces, with attrition. Previously reported data on the bond strength to fluorosed enamel in patients below and above the age of 40 years show a difference.[14] Therefore, sample teeth used were obtained from patients of 20 to 40 years of age.

Patient demand for the treatment of unesthetic anterior teeth has grown. Since the introduction of porcelain laminate veneers, their popularity has increased because tooth preparation is conservative and the restorations are esthetic.[15] Similarly, fluorosed teeth are usually restored with tooth-colored restorations such as composite resins or ceramic veneers.[16] The success of a porcelain veneer is greatly determined by the strength and durability of the formed bond between the three components of the bonded veneer complex, namely the tooth surface, the luting composite, and the porcelain veneer.[17]

For microtensile testing, the tensile bond strength is dependent on the cross-sectional area of the bonded surface. When the beam method of specimen preparation is employed, the MTBS test offers a further advantage over conventional tensile tests. Multiple beams can be harvested from a single specimen, and since these beams are not trimmed before testing, there is less stress on adhesive bonds.[18] In this study, failures occurred at the bonding resin–enamel interface and did not involve the enamel or ceramic except for some specimens, which showed cohesive failure within the ceramic. Microtensile testing should more closely approximate clinical applications; however microcracks and other defects can possibly occur

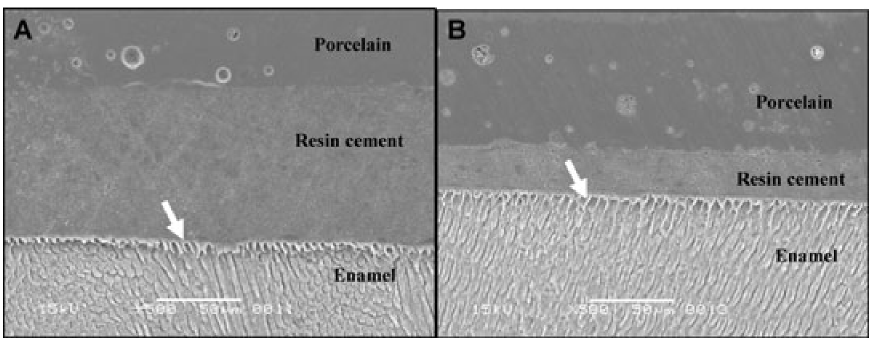

Figure 3 SEM images of adhesive interface of porcelain veneer bonded to fluorosed (A) and normal (B) enamel with RelyX showing about 7 μm of resin tag-like penetrations in the enamel (arrows).

during the preparation of specimens with a slow-speed diamond saw sectioning machine, which may cause premature failure of the bond. Therefore the specimens must be prepared carefully.

Current ceramic bonding systems are based on micromechanical binding between cement and ceramic restorations. Porcelain surface preparations for mechanical retention include grinding, sandblasting, and etching with acids. The application of silane couplers served as the chemical surface preparation for bonding porcelain. The use of silane primers considerably enhanced the bond strength of porcelain.[19]

Al-Sugair and Akpata[20] reported that the etching time of moderately fluorosed teeth should be doubled to get typical etching patterns. Similarly, for severely fluorosed teeth, the hypermineralized surface layer should be ground away before etching the subsurface enamel, and the etching time should be prolonged by at least about 90 seconds. Ateyah and Akpata[21] reported similar bond strengths of ground enamel to composite resin for all groups of fluorosis in young patients. They attributed this similarity to the loss of hypermineralized surface layer due to the use of ground enamel as bonding substrate. Moreover, they reported that bonds tend to fail predominantly cohesively in the enamel when the fluorosis was severe. This difference in fracture pattern was not observed in our study, as unequal stress distribution resulting in cohesive failure in enamel can be avoided with the MTBS test.

In the present study, VITA VM7 porcelain laminate veneers were bonded with two resin cement systems, RelyX and Clapearl, to fluorosed and normal enamel. Both the resin cement systems used in this study contained silane-coupling agents and used an acid on the porcelain and enamel surface for micromechanical retention. The procedure for bonding of porcelain laminate veneers to both normal and fluorosed enamel was the same as that instructed by the manufacturer of the materials used. Extended etching times and special treatments were not followed for fluorosed teeth as suggested in previous reports.[22] The adhesive systems exhibited no significant differences in MTBS between the two groups ($p > 0.05$). The two resin cement systems demonstrated strong bonding to the porcelain laminate veneers and to normal and fluorosed enamel. This result indicates that enamel bond strength of both the adhesive systems was not affected by the moderate fluorosis; however, according to a previous study on fluorosed teeth, bonding systems that use phosphoric acid etching with an extended time of 90 seconds may be more suitable for bonding fluorosed enamel.[23] But according to the results of this study, even though longer etching times were not used, there were no statistically significant differences in bond strengths of normal and fluorosed enamel. Therefore, the hypothesis that the type of enamel affects the MTBS of porcelain laminate veneers must be rejected. Furthermore, any extra treatment such as extended etching times or special preparations may not be needed when veneers are bonded to fluorosed teeth.

This study was designed to simulate the exact clinical conditions of adhesive bonding of porcelain laminate veneers in vital teeth. Although severely fluorosed teeth would have been ideal for this study (as the clinical procedure explained was most suitable for those teeth), difficulty in obtaining such teeth limited optimizing the final result.

Conclusions

Within the limitations of this study the following conclusions were drawn:

1. There were no differences in enamel bonding strength between RelyX and Clapearl cement systems.
2. The MTBSs of two resin ceramic cement systems to porcelain laminate veneers and to fluorosed enamel were not influenced by the severity of fluorosis.
3. Therefore, extra treatment or special preparations are not needed when veneers are bonded to fluorosed teeth.

Acknowledgments

The authors wish to thank Dr. K. A. Wettasinghe of the Faculty of Dental Sciences, University of Peradeniya, Sri Lanka, for his contribution in collecting fluorosed teeth for this project. We also extend our sincere gratitude to Dr. Kouji Inoue of the Institute of Electron Microscopy of Tsurumi University School of Dental Medicine.

References

1. Aoba T, Fejerskov O: Dental fluorosis: chemistry and biology. Crit Rev Oral Biol Med 2002;13:155-170
2. Fejerskov O, Richards A, Denbesten P: Fluoride in Dentistry (ed 2). Copenhagen, Denmark, Munksgaard, 1996, pp. 112
3. Dissanayake C: Of stones and health: medical geology in Sri Lanka. Science 2005;309:883-885
4. Lim CC: Case selection for porcelain veneers. Quintessence Int 1995;26:311-315
5. Meyer Filho A, Vieira LC, Baratieri LN, et al: Porcelain veneers as an alternative for the esthetic treatment of stained anterior teeth: clinical report. Quintessence Int 2005;36:191-196
6. Akpata ES: Occurrence and management of dental fluorosis. Int Dent J 2001;51:325-333
7. Aristidis GA, Dimitra B: Five-year clinical performance of porcelain laminate veneers. Quintessence Int 2002;33:185-189
8. Fiedman MJ: A 15 year review of porcelain veneer failure, a clinician's observations. Compendium 1998;19:625-636
9. Thylstrup A, Fejerskov O: Clinical appearance of dental fluorosis in permanent teeth in relation to histological changes. Community Dent Oral Epidemiol 1978;6:315-328
10. Fejerskov O, Manji F, Baelum V: Dental Fluorosis—A Handbook for Health Workers. Copenhagen, Denmark, Munksgaard, 1988, pp. 45
11. Osuji OO, Leake JL, Chipman ML, et al: Risk factors for dental fluorosis in fluoridated community. J Dent Res 1988;67:1488-1492
12. Pang DTV, Phillips CL, Bowden JW: Fluoride intake from beverage consumption in a sample of North Carolina children. J Dent Res 1992;71:1382-1388
13. Ekanayake L, Hoek W: Prevalence and distribution of enamel defects and dental caries in a region with different concentration of fluoride in drinking water in Sri Lanka. Int Dent J 2003;53:243-248
14. Ateyah N, Akpata E: Factors affecting shear bond strength of composite resin to fluorosed human enamel. Oper Dent 2000;25:216-222
15. Nash RW: Esthetic restoration of discolored teeth using porcelain laminate veneers. Compendium 1998;19:518-538

16. Schuman I: Simplified restorative correction of the dentition using contact lens-thin porcelain veneers. Dent Today 2006;25:88-92
17. Usumez A, Aykent F: Bond strengths of porcelain laminate veneers to tooth surfaces prepared with acid and Er,Cr:YSGG laser etching. J Prosthet Dent 2003;90:24-30
18. Aykent F, Usumez A, Ozturk AN, et al: Effect of provisional restorations on the final bond strengths of porcelain laminate veneers. J Oral Rehabil 2005;32:46-50
19. Stacey GD: A shear stress analysis of the bonding of porcelain veneers to enamel. J Prosthet Dent 1993;70:395-402
20. Al-Sugair MH, Akpata ES: Effect of fluorosis on etching of human enamel. J Oral Rehabil 1999;26:521-528
21. Ateyah N, Akpata ES: Factors affecting shear bond strength of composite resin to fluorosed human enamel. Oper Dent 2000;25:216-222
22. Weerasinghe DS, Nikaido T, Wettasinghe KA, et al: Micro-shear bond strength and morphological analysis of a self-etching primer adhesive system to fluorosed enamel. J Dent 2005;33:419-426
23. Jayasooriya PR, Wettasinghe KA, Ogata M, et al: Micro-tensile bond strength of a self-etching primer bonding system to fluorosed enamel. Int Chin J Dent 2002;2:107-115