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Original article

Evaluation of the effect of surface preparation using phosphoric acid and luting cement on the flexural strength of porcelain laminate veneering material

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ABSTRACT

Background: Conventionally HF acid has been used for etching ceramic veneer restorations before their cementation. Studies are lacking regarding the effectiveness of phosphoric acid as a substitute for HF acid for etching the ceramic veneers. The purpose of this study was to evaluate the effectiveness of surface preparation of porcelain laminate veneers using phosphoric acid, as compared to HF acid etching in providing the necessary surface roughness conducive to development of an effective bond between the ceramic laminate and the resin luting cement.

Methods: 210 porcelain discs of 15 mm diameter and 0.9 mm thickness were prepared. These study samples were divided into seven groups of thirty samples each. Surfaces of the first (control) and the second group of samples were not prepared. The surfaces of other five groups were prepared with different surface treatments. Further all the groups of specimens were coated with a layer of resin luting cement. Flexural strength of each specimen was determined using universal testing machine and the results were compared. **Results:** The combination surface treatment using alumina surface abrasion followed by etching with phosphoric acid provided the highest flexural strength with the mean flexural strength of 101.11 MPa, followed by alumina surface abrasion (95.41 MPa), and phosphoric acid surface etching (81.68 MPa).

Conclusion: Laminate veneers surface treated using 50 μ m alumina abrasion followed by etching with phosphoric acid showed the highest flexural strengths after resin coating compared to other groups.

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Introduction

Porcelain laminate veneers have been used extensively and successfully to improve anterior tooth esthetics. The

popularity of these restorations is attributable to their esthetic quality, conservative preparation, fracture resistance, tissue acceptance, low debonding rate, patient satisfaction, and negligible incidence of caries.

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The clinical success of porcelain laminate veneer restorations is dependent on the intimate bond achieved between the restoration and tooth structure obtained via the resin cement.¹ The factors known to affect the effectiveness of resin/ceramic bond include the method of surface preparation using aluminum oxide surface abrasion² and etching with hydrofluoric acid.³ These procedures create micro-irregularities on the porcelain surface, which help in providing a mechanical interlock with composite resin. This physical bond combines with the chemical bond obtained from the use of a silane coupling agent to provide a durable bonding effect between the etched porcelain and resin luting cement. Effective etching of the porcelain surface is considered to be an essential step for a successful restoration. Thus a study of morphologic patterns of etched porcelains, with various etchants, should yield information vital to the clinical success of etched porcelain bonded restorations.

The most commonly used etchant is 5% solution of hydrofluoric acid (HF acid). HF solution is considered to be hazardous to health and requires isolated workplaces with adequate ventilation. Studies regarding the efficiency of phosphoric acid as a viable alternative to etch ceramic are lacking.

Clinical failure of porcelain laminate veneer restorations is thought to be due to development of flaws on the surface of the restorations.⁴ Surface imperfections act as a potential source of crack propagation which may be either inherent in the porcelain or introduced during the porcelain laminate veneer fabrication, surface treatment or cementation. Post-operative failure of the restoration also occurs as a consequence of thermal, mechanical and chemical variations that these restorations are likely to encounter in their service.⁵ Further the resin cement used for luting the laminate veneer may impose surface changes on the veneer when subjected to thermocycling.

The purpose of this study is to evaluate the effectiveness of surface preparation of porcelain laminate veneers made up of

low-fusing feldspathic porcelain using phosphoric acid, as compared to HF acid etching and aluminum oxide surface abrasion in providing the necessary surface roughness conducive to development of an effective bond between the ceramic laminate and the resin luting cement.

Materials and method

The materials and method followed in this study is as follows:

Fabrication of ceramic disc specimen

About 0.6 g of Vitadur-alpha dentin powder was pre-weighed in an electronic balance and 0.22 ml of modeling fluid measured in micropipette. Ceramic powder was mixed with modeling fluid, placed in the custom made metallic mold and compacted. Discs were placed on the platinum sheet and then fired in Multimat vacuum furnace (Vita) according to manufacturer's instructions. 210 ceramic test specimens of 15 mm diameter and 0.9 mm thickness were fabricated.

Grouping of the test specimens

The specimens were divided into seven groups of thirty specimens each (Fig. 1). They were designated as group A–G.

Surface treatments of test specimens

The unglazed surface of specimen was surface treated as designated under:

1. **Group A (Control)** – no surface treatment.
2. **Group B** – no surface treatment, but the samples were thermocycled.
3. **Group C** – samples were surface treated using alumina abrasion by keeping them 2 cm away from the nozzle of the

Fig. 1 – Grouping of the ceramic samples into seven different groups.

sandblaster using 50 µm aluminum oxide particle at 70 psi pressure for 5 s.

4. **Group D** – samples were etched with 5% hydrofluoric acid for 90 s.
5. **Group E** – samples were etched with 37% phosphoric acid (Fig. 2) for 5 min.
6. **Group F** – samples were surface treated with 50 µm aluminum oxide surface abrasion for 5 s at a distance of 2 cm at 70 psi pressure followed by etching with 5% hydrofluoric acid for 90 s.
7. **Group G** – samples were surface treated with aluminum oxide surface abrasion for 5 s at a distance of 2 cm at 70 psi pressure followed by etching with 37% phosphoric acid for 5 min.

Observation of the surface appearance under SEM

The prepared surfaces of ceramic specimen (Group A–Group G) were examined under scanning electron microscope for qualitative surface appearance, at magnification of 3000×.

Coating the ceramic disc specimens with resin luting cement

Prior to cementation of the surface of the disc specimen surface was primed for resin using Monobond S (Vivadent, NY, USA), for 60 s and air dried before cementation. Resin cement Multilink Automix, Schaan/Liechtenstein (Fig. 3) was used for the luting the test specimens. The mixed cement was placed on a glass on cover slip; the porcelain discs were pressed lightly and measured with a micrometer to achieve a film thickness of 50 µm. The excess cement was expressed. The resin cement was light activated for 40 s to initiate the polymerization reaction.

Thermocycling procedure

The samples (except Group A) were embedded in silicone putty impression material loaded in a mesh tray to expose only the glazed surface and thermocycled between the maximum and the minimum temperature $65 \pm 1^\circ\text{C}$ – $4 \pm 1^\circ\text{C}$.

Fig. 2 – Phosphoric acid (37%) for the surface preparation of ceramic.

Fig. 3 – Multilink Automix resin cement.

The tray was transferred between water baths with a 5 s dwell time in each water bath and a transfer time of 70 s for a complete cycle and 3500 such cycles were carried out.

Evaluation of flexural strength of laminate veneers

A ball on ring test was employed to assess the fracture strength of the surface prepared specimens using a Universal testing machine (Hounsfield) (Fig. 4). A loading ring apparatus with a 10 mm diameter knife edge support and a spherical ball indenter of 4 mm diameter at a strain rate of 1 mm/min was used to test the flexural strength of the specimen. The bi-axial fracture strength was calculated according to the following equation.

$$\sigma_{\text{Max}} = P/h^2 \{1 + \nu[0.485 \ln(a/h) + 0.52] + 0.48\}$$

Statistical analysis

The student's t-test was used for the statistical analysis to determine the difference between the strength values of the different groups.

Fig. 4 – Universal testing machine breaks the test specimen using a custom made fixture.

Results

The flexural strengths of specimens along with the mean and the standard deviation are given in Table 1. The combination surface treatment using alumina surface abrasion followed by etching with phosphoric acid provided the highest flexural strength with the mean flexural strength of 101.11 MPa, followed by alumina surface abrasion (95.41 MPa) and phosphoric acid surface etching (81.68 MPa). The surface preparation using HF acid etching provided a mean flexural strength of 70.14 MPa. The lowest strength was given by surface treatment using alumina surface abrasion followed by etching with HF acid (65.37 MPa).

Scanning electron microscope (SEM) observations

The observation of specimen under scanning electron microscope revealed the following pictures (Fig. 5):

Group A: samples of Group A showed smooth surface without irregularities on the test surface of specimen.

Group B: the samples showed a few porosities and cracks on their surface.

Group C–G: samples of Group C–G showed irregular surfaces with occasional channels/groves extending to the deeper side of the specimen. Specimen abraded by alumina (Group C) had a uniform irregular abraded surface where as the specimen etched by both HF acid (Group D) and phosphoric acid (Group E) showed irregular etched surfaces with a few occasional unetched areas. Specimen surfaces etched by HF acid had deeper groves than those etched by phosphoric acid.

Discussion

The bonding of composite to ceramic materials has played an important role in the success of laminate restorations. Surface preparation of laminate veneering material is important for the strong resin and the ceramic bond. Factors known to improve the effectiveness of resin/porcelain bond include the method of surface preparation namely aluminum oxide surface abrasion² and hydrofluoric acid (HF) etching.³ The retentive qualities of the porcelain surface will depend upon the nature of microscopic pattern produced during the surface

Table 1 – Showing the complete data on flexural strength of specimens along with mean and the standard deviation.

Sample No	Surface treatment						
	Control	Thermocycling	Alumina abrasion	HF acid etching	Phosphoric acid etching	Alumina + HF acid	Alumina + Phosphoric acid
1	93.65	61.08	96.48	69.96	90.22	56.42	103.92
2	102.32	63.36	95.82	64.91	82.21	63.55	107.74
3	95.56	58.54	95.98	65.93	78.14	59.43	79.93
4	90.45	59.13	92.53	69.63	80.32	64.98	97.82
5	89.82	62.14	92.46	74.11	78.62	66.53	103.48
6	93.56	64.16	121.22	65.72	69.54	61.24	98.74
7	78.34	70.92	90.92	59.92	82.44	68.92	113.96
8	96.34	61.56	79.88	67.52	72.12	63.4	93.76
9	90.37	71.84	89.92	68.83	83.46	70.46	99.95
10	91.92	68.74	95.94	75.94	82.36	62.34	102.03
11	92.69	63.71	97.59	58.89	79.96	56.39	89.93
12	92.45	57.44	103.94	68.85	77.98	69.79	108.07
13	102.6	65.65	92.32	66.72	81.34	82.46	99.79
14	88.54	68.59	93.56	64.96	83.63	59.74	97.49
15	96.9	58.44	101.19	74.92	81.52	78.94	115.08
16	101.34	60.92	105.38	67.32	78.12	56.86	95.94
17	89.98	67.59	97.05	75.74	82.34	66.74	99.98
18	89.64	52.35	98.65	68.95	81.86	64.52	109.29
19	88.34	53.08	95.39	82.19	69.94	70.42	103.85
20	86.65	61.53	89.96	74.46	82.31	62.76	99.46
21	90.98	57.67	94.65	84.14	110.32	68.98	104.42
22	96.98	49.86	95.96	73.76	81.83	66.42	97.92
23	89.37	52.91	89.91	68.81	83.85	48.93	99.48
24	92.33	61.67	91.67	63.77	79.64	72.87	102.17
25	76.87	55.88	108.56	65.94	81.42	78.08	105.09
26	93.78	48.32	92.46	72.82	90.28	65.48	97.18
27	91.86	79.09	78.91	77.66	83.65	58.86	103.92
28	104.77	64.8	91.46	69.92	80.56	74.38	98.82
29	89.98	53.24	97.04	68.86	78.98	51.84	100.96
30	93.44	63.17	95.48	72.96	81.38	69.48	103.18
Mean	92.39	61.25	95.41	70.14	81.68	65.37	101.11
SD	6.04	6.90	7.79	5.77	7.02	7.72	6.71

Fig. 5 – SEM picture showing the sample after surface preparation with phosphoric acid at 3000× magnification.

preparation. Brittle materials such as dental ceramics are susceptible to fracture under the impact of stresses produced at the surface defects that are either inherent of the material and/or introduced during processing.^{6–8} Ceramics are susceptible to slow crack propagation at the tips of the surface flaws exposed to moist environment as a result of hydrolysis of silicate bonds⁹ resulting in decreased strength of the material.¹⁰

Further, surface flaws may become extended due to thermal variation induced by the ingested foods and drinks is an important aspect which has to be considered for any restoration.

According to manufacturer's instructions Vitadur-Alpha dentin power 0.6 g was condensed with 0.22 ml of Vita modeling fluid to get an optimum slurry consistency for producing this series of nominally identical dentine porcelain disc specimens. Studies have shown that this consistency will lead to decrease in porosity and an increase in the solid density.¹¹ Thermocycling procedure was carried out at the maximum and minimum temperatures of $65 \pm 1^\circ\text{C}$ and $4 \pm 1^\circ\text{C}$ to simulate the temperature changes of oral cavity.

Fig. 6 – Showing the mean strengths vs treatments.

This is in accordance with the study by Palmer et al¹² temperature extremes will have an effect on the longevity of porcelain laminate restorations. In the oral environment high and low temperatures are only transient, and to simulate the clinical situation, a short dwell time (5 s) was used in the study. Three thousand five hundred thermocycles for each test sample was chosen to simulate the thermal changes happening in the oral cavity for a period of one year assuming that at most 10 extreme thermocycles would occur in a day.¹²

Effective and most commonly used surface preparation methods indicated by different authors are alumina surface abrasion¹³ and etching with hydrofluoric acid.³ Studies have also shown that amongst different sizes, 50 µm alumina provides the best surface modification conducive to ceramic-resin cement bonding.¹⁴ Hydrofluoric acid solutions are considered to be hazardous to health and require isolated workplaces with adequate ventilation. Due to this reason products labeled as hydrofluoric acid substitutes are preferred by some technicians and dentists. Phosphoric acid has been used as substitute for the surface preparation of porcelain laminate veneers by many authors. Bond strength achieved with the resin cement to ceramic after surface preparation using phosphoric acid has been found to be almost equal to that done with hydrofluoric acid.^{5,8,15} John W Thurmond et al showed that phosphoric acid is inferior compared to HF acid in creating the micro-irregularities for the effective bonding between the ceramic surface and resin cement.¹⁵ There is no data available about the effect of etching of ceramic surface with phosphoric acid on the survival probability of laminate veneers.

Results of this study showed that the HF acid etched specimen (Group D) showed increased strength compared to thermocycled group (Group B), but their strength was lower when compared to control group (Group A) and the alumina abrasion group (Group C). This shows that although etching with a strong acid like hydrofluoric acid produces mechanical irregularities for effective bonding but it also had the weakening effect on ceramic surface probably because of the surface acidity as reported by Addison and Flemming.¹⁶

Results of this study also showed that the mean strength of phosphoric acid etching group (81.68 MPa) was significantly ($p \leq 0001$) lower than that of the control group (92.39 MPa) (Fig. 6) and was also less than the alumina abrasion group. Flexural strength of the specimen which was treated with phosphoric acid is higher than the HF acid surface preparation group (Group C). This showed that etching with phosphoric acid had a less damaging effect on the ceramic surface compared to HF acid.

The comparison between control group (Group A) and the group which was treated with combination surface treatment with alumina and HF acid (Group F) showed that the mean strength of the later group (65.37 MPa) was significantly lower than that of former. Although apparently the mean strengths of alumina + HF acid group was higher than mean strengths of thermocycling group by only 4 MPa, the difference was statistically significant (Table 2). Alumina + phosphoric acid group specimens (Group G) (101.11 MPa) were significantly better than all other groups. The difference between the flexural strength of control group (Group A) and alumina + phosphoric acid surface treatment group (Group G) was also statistically significant.

Table 2 – Paired comparisons using test with 't' values and p-values in the parentheses.

Treatments	Thermocycling	Alumina	HF acid	Phosphoric acid	Alumina + HF	Alumina + Phosphoric acid
Control	18.60***, * (0.0001)	–1.68 NS (0.099)	14.60**** (0.0001)	6.34**** (0.0001)	15.10**** (0.0001)	–5.29**** (0.0001)
Thermocycling		–17.98**** (0.0001)	–5.41**** (0.0001)	11.37**** (0.0001)	–2.18* (0.033)	–22.68**** (0.0001)
Alumina abrasion			14.28**** (0.0001)	7.17 **** (0.0001)	15.00**** (0.0001)	–3.04** (0.004)
HF Acid Etching				–6.96**** (0.0001)	2.71** (0.009)	–19.18**** (0.0001)
Phosphoric Acid					8.56**** (0.0001)	–10.96**** (0.0001)
Alumina + HF						–19.13**** (0.0001)

NS: not significant $p > 0.05$.*: Significant: $p \leq 0.05$.**: Highly significant: $p \leq 0.01$.***: Very highly significant: $p \leq 0.001$.****: Very very highly significant: $p \leq 0.0001$.

The best surface treatment was alumina + phosphoric acid group (Group G) (101.11 MPa) followed by alumina surface abrasion group (Group C) (95.41 MPa) and phosphoric acid surface etch group (Group E) (81.68 MPa).

The mean strengths of all surface treated and thermocycled samples were more than thermocycled control samples. This showed that surface preparation method used to create the bonding between ceramic and resin cements modify the surface flaws present in the ceramic. Polymerization shrinkage of resin cement probably causes a layer of compressive stress on ceramic layer leading to reduction in the potential of crack propagation by healing the surface flaws and also increasing the strength of ceramic.¹⁶

Ceramic surface treatment with 37% phosphoric acid provides necessary surface for the resin bonding, but required long etching time of 5 min compared to hydrofluoric acid, which required only 90 s.

Summary and conclusion

Within the scope of the study the following conclusions are drawn:

1. All surface preparation methods used in this study i.e. alumina abrasion, etching with HF acid and 37% phosphoric acid produced an effective surface preparation of specimen which was conducive for micromechanical bonding between the ceramic specimen and the resin cement.
2. Flexural strength of untreated ceramic specimen reduced after thermocycling procedure.
3. The surface treatment of the ceramic specimen using 37% phosphoric acid increased their flexural strength when luted with resin cement layer even after thermocycling procedure. So phosphoric acid (37%) alone or in combination with 50 μm alumina abrasion can be used for the surface preparation of ceramic laminate veneers before bonding them to tooth surface to improve their longevity.

Conflicts of interest

All authors have none to declare.

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