Effect of phosphoric acid and sodium hydroxide on cleaning and bonding of saliva-contaminated feldspar porcelain

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Abstract

Purpose: Hydrofluoric acid has been used to remove salivary contamination in dental glass-ceramics before bonding treatment. However, alternative methods are required because hydrofluoric acid is harmful. This study examined the cleaning effects of phosphoric acid and sodium hydroxide on glass-ceramics for bonding pre-treatment.

Methods: Feldspar porcelain was divided into four groups: (C) cleaned porcelain without any contamination, (S) porcelain contaminated with saliva, (SPA) porcelain cleaned with 37% phosphoric acid after saliva contamination, and (SSH) porcelain cleaned with 10% sodium hydroxide after saliva contamination. Each sample was bonded to the resin cement using a silane-containing primer. They were then subjected to a shear bond strength (SBS) test. Each surface was analyzed by scanning electron microscopy (SEM), contact angle measurements, and Fourier transform infrared spectroscopy (FT-IR).

Results: The SBS of group SSH was comparable to that of group C but significantly higher than that of groups S and SPA. SEM observations showed that saliva-like structures remained on the samples of groups S and SPA, but not on the SSH group. The contact angles of groups C and SSH were comparable and significantly smaller than those of groups S and SPA, respectively. FT-IR analysis also revealed saliva in groups S and SPA, which was absent in the SSH group.

Conclusion: The saliva remained on the porcelain even after cleaning with phosphoric acid, and SBS was not restored to the same level as before the contamination. In contrast, sodium hydroxide eliminated saliva and restored SBS to the same level as before contamination.

Keywords: Glass-ceramics, Porcelain, Alkali, Acid, Shear bond strength

1. Introduction

Glass-ceramics such as feldspar porcelain and lithium disilicate glass are used in tooth restorative materials, including inlays, crowns, and laminate veneers, because of their excellent esthetics and biocompatibility [1-3]. Clinical problems associated with the use of glass-ceramics for tooth restoration include fracture and debonding failure [4]. These failures are influenced by the bonding of glass to glass-ceramics [5-8]. Thus, the use of appropriate bonding procedures is critical for successful long-term glass-ceramic restorations.

Among the clinical methods for the bonding pre-treatment of glass-ceramic crowns, a combination of cleaning and etching using hydrofluoric acid and priming the glass-ceramic surface using a silane coupling agent is commonly used [9-12]. Cleaning and etching of glass-ceramic crown with hydrofluoric acid are performed under two conditions: chair-side at dental clinics by a dentist or in a dental laboratory by a dental technician [13]. In the chair-side case, the glass-ceramic crown is treated with hydrofluoric acid to eliminate saliva and etch the glass-ceramic surface after it has been contaminated with saliva during trial fitting of an abutment tooth in the patient’s oral cavity. In contrast, in dental laboratories, the glass-ceramic crown is treated once with hydrofluoric acid immediately after fabrication to etch the glass-ceramic surface. The dentist then uses the pre-treated glass-ceramic crown in the trial, fitting the chair-side with the patient. The resultant glass-ceramic crown, contaminated with saliva, will be cleaned with hydrofluoric acid or another reagent. In these bonding processes, salivary contamination on the surface of the glass-ceramic crown is considered to hinder the bonding between the ceramic and the luting agent. However, the complete elimination of salivary contaminants by a cleaning process, particularly the removal of salivary proteins, is challenging [14-16]. Cleaning only with water and no other reagents does not sufficiently eliminate salivary contamination [17-19]. Therefore, hydrofluoric acid treatment is recommended as an indispensable step after cleaning with water [16,20]. Nevertheless, hydrofluoric acid is extremely toxic to the human body and must be used with caution [21]. Moreover, if possible, the use of hydrofluoric acid in dental clinics should be avoided because it is risky for the operator. In dental clinics, alternative treatment methods for eliminating salivary contamination that do not use hydrofluoric acid are required.

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Table 1. Materials used in the present study

| Product name     | Material type          | Manufacturer         | Composition                                                                 |
|------------------|------------------------|----------------------|----------------------------------------------------------------------------|
| Vitablocs Mark II| Glass ceramic          | VITA Zahnfabrik H. Rauter GmH & Co.KG, Germany | Silicon dioxide 56-64%, Aluminum oxide 20-23%, Sodium oxide 9-11%, Potassium oxide 6-8%, Calcium oxide 0.3-0.6%, Titanium dioxide 0.0-0.1%* |
| Super-Bond PZ Primer | Silane coupling agent | Sun Medical Inc., Japan | Liquid A: Methacrylic monomer, etc. Liquid B: Methacrylic monomer, Silane coupling agent, etc. |
| ResiCem PASTE | Dualpolymerized resin cement | Shofu Inc., Japan | Paste A: UDMA, TEGDMA, Fluoroaluminosilicateglass, Initiator, etc. Paste B: UDMA, TEGDMA, Fluoroaluminosilicateglass, 4AET, HEMA, Fluoroaluminosilicateglass, Initiator, etc. |

*The composition of Vitablocks Mark II refer to the study [34].

2. Materials and Methods

2.1. Materials

The materials used in this study are listed in Table 1. Initially, the Vitablocs Mark II was cut to a thickness of 1 mm using a diamond cutter, resulting in a sample with dimensions of 8 mm x 8 mm x 1 mm. The cut samples were polished with #600 SiC paper and the thickness was adjusted to ±0.1 mm. The samples were ultrasonically cleaned with distilled water for 5 min, followed by drying in the air. The samples were randomly divided into the following four groups:

- Group C: Porcelain samples that were neither contaminated with saliva nor treated (control group).
- Group S: Porcelain samples that have been immersed in saliva collected at 37 °C for 60 s to achieve salivary contamination and then allowed to air-dry completely to promote saliva adhesion onto the porcelain surface. Contaminated samples were then washed with water for 15 s and air-dried. The saliva used in the experiments was collected from a healthy adult male who had not consumed any solid or liquid for 2 h. Saliva was collected in a beaker and used immediately for the experiment, as described in a previous study [24]. The experimental protocol using human saliva was approved by the ethics committee of our university (approval number 19-85).
- Group SPA: The porcelain samples were contaminated with saliva and then dried to enable adhesion of the saliva as in group S. To remove salivary contamination, the contaminated samples were immersed in 37% phosphoric acid for 2 min, followed by 15 s of washing with water and subsequent drying using an air blower. An aqueous solution of phosphoric acid (hereafter phosphoric acid) used for cleaning was prepared in our laboratory by diluting 85 w/v% phosphoric acid (Fujifilm Wako Pure Chemical Corp., Japan) with distilled water and mixing it with a magnetic stirrer to adjust it to a concentration of 37 w/v%.
- Group SSH: The porcelain samples were contaminated with saliva and then dried to enable adhesion of the saliva as in group S. To remove salivary contamination, the contaminated samples were then immersed in 10% aqueous solution of sodium hydroxide (hereafter sodium hydroxide) (Fujifilm Wako Pure Chemical Corp., Japan) for 2 min, followed by 15 s of water washing and subsequent drying using an air blower as for group SSH. The concentration of sodium hydroxide was determined based on the results of a previous study on the prion cleaning of dental nickel-titanium files [29].

2.2. Shear bond strength test

The shear bond strength (SBS) between the porcelain and resin cement was determined using 10 samples from each of the four groups (n = 10). Teflon tubes with 5 mm inner diameters were fixed to each sample using tape to achieve a constant bonding area. The PZ primer and liquids A and B were mixed according to the manufacturer’s instructions, and the resultant mixture was applied to each sample using tape to achieve a constant bonding area. The PZ primer and liquids A and B were mixed according to the manufacturer’s instructions, and the resultant mixture was applied to the sample surface. After being maintained under ambient conditions for 60 s, the surface was dried using an air blower. The resin cement was loaded on the sample surface at a 3-mm-height. Subsequently, the cement-loaded samples were subjected to light irradiation for 5 min using a light irradiator (α LIGHT II N, J. Morita Corp., Tokyo, Japan) with saliva nor treated (control group).

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to cure the resin cement. To ensure the curing of the resin cement, we used a light irradiator instead of a handheld light-curing unit. The Teflon tube and fixing tape were removed from the samples, and the samples were maintained at 25 °C for 30 min under ambient conditions. The samples were immersed in water at 37 °C for 24 h. The resultant samples were subjected to an SBS test using a mechanical test machine (AGS-H, Shimazu Corp., Japan) at a crosshead speed of 1.0 mm/min. After testing, the cement-debonded surfaces were observed using an optical microscope and classified into the following failure modes: A, adhesive failure at the cement-porcelain interface; C, cohesive failure within the porcelain; and M, mixed failure (A and C).

2.3. Surface characterization

The cleaning effects on saliva-contaminated porcelain samples were characterized by Fourier transform infrared spectroscopy (FT-IR), contact angle tests, and scanning electron microscopy (SEM).

The IR spectrum of the surface of each sample was recorded using an FT-IR spectrometer (IRSpirit, Shimadzu Corp. Japan) equipped with an attenuated total reflection unit in the wavenumber range of 1300-2000 cm⁻¹ with a resolution of 4 cm⁻¹. According to a previous study [30], the characteristic peak attributed to N-H bending coupled with C-N stretching appeared at 1632 cm⁻¹. The IR spectrum of the surface of each sample was recorded using an FT-IR spectrometer (IRSpirit, Shimadzu Corp. Japan) equipped with an attenuated total reflection unit in the wavenumber range of 1300-2000 cm⁻¹ with a resolution of 4 cm⁻¹, according to a previous study [30].

The water contact angle of each sample was measured at 20 °C to determine the surface wettability, using a contact angle meter (DMe-211, Kyowa Interface Science Co., Ltd. Japan). A 2-μL droplet of distilled water was dropped on the sample surface and kept for 5 s, and its image was captured by the camera of the contact angle meter. Measurements were performed on 10 samples among each group (n =10).

For the SEM observation, each sample was coated with platinum using a sputtering device. The sample was examined with a scanning electron microscope (JCM-7000, JEOL Ltd., Japan) in backscattering mode at an acceleration voltage of 10 kV and a working distance of 15 mm.

2.4. Statistical analysis

The results of the SBS and contact angle measurements were statistically analyzed using EZR statistical software (Saitama Medical Center, Jichi Medical University, Japan). The Kolmogorov-Smirnov test was primarily used to check the data distribution. The results did not show a normal distribution. Therefore, non-parametric multiple comparisons were performed using the Kruskal-Wallis test followed by the Steel-Dwass test. The significance level was set at P < 0.05.

3. Results

Table 2 lists the results of the SBS test, and Table 3 lists the failure modes of the samples after the SBS test. The SBS of group S was significantly lower than that of group C, and the failure mode of all samples was adhesive failure. The SBS score of the SSH group was comparable to that of group C, and its failure mode showed both cohesive and mixed failures. The SBS of the SPA group was significantly lower than the groups C and SSH. Simultaneously, the SBS of the SPA group was significantly higher than that of group S, and the failure mode of most of the samples was adhesive failure.

The SEM results are shown in Figure 1. No salivary contamination was observed in samples from groups C and SSH. In contrast, the sample in group S was covered with saliva-like structures over the entire surface. In the SPA group sample, the surface was partially covered by saliva-like structures. The results of the contact angle tests are presented in Table 4. There was no significant difference in the contact angles between groups C and SSH, or between groups S and SPA. The contact angles of groups S and SPA were significantly higher than those of groups C and SSH. The FT-IR analysis results are shown in Figure 2. In the spectra of groups S and SPA, the characteristic peak attributed to N-H bending coupled with C-N stretching
Table 4. Mean values and standard deviations (SD) for contact angles of distilled water on each group of porcelain (C: porcelain without salivary contaminants; S: porcelain contaminated with human saliva; SPA: porcelain cleaned by 37% phosphoric acid after contamination with human saliva; SSH: porcelain cleaned by 10% sodium hydroxide after contamination with human saliva). The different letters in the category indicate significant differences between the sample groups (p<0.05, Steel-Dwass’s test, n=10).

| Sample groups | Contact angle | Category |
|---------------|---------------|----------|
| C             | 27.5 (3.8)    | a        |
| S             | 50.8 (5.6)    | b        |
| SPA           | 42.3 (14.9)   | b        |
| SSH           | 24.1 (3.6)    | a        |

![FT-IR spectra of each group of porcelain](image)

The present experiments were performed after the saliva had completely dried and adhered to the porcelain surface to simulate the most serious situation in clinical practice, where the adhered saliva inhibits bonding with the resin cement. From the SEM observations, it was inferred that salivary contamination in this study was more severe than that in the previous studies [24,25]. Salivary contamination remained on the sample surface and substantially reduced the SBS between porcelain and resin cement. This is consistent with the results of previous reports showing degradation of bonding between ceramics and resin cement in the presence of saliva [15,16,31]. In studies on salivary cleaning with agents, the application time for the cleaning of saliva is often 30 s [25,26]. In our preliminary experiment, however, the cleaning effects of phosphoric acid or sodium hydroxide were not evident at such a short application time because completely dried saliva adhered strongly to the porcelain surface. Therefore, the application time was prolonged to 120 s. This prolongation revealed the cleaning effects of phosphoric acid and sodium hydroxide on adhered salivary contaminants.

SEM observations indicated that salivary contamination was present on the porcelain surfaces of groups S and SPA, but not on the surface of group SSH. In addition, the contact angle of group SSH was comparable to that of group C and significantly lower than that of groups S and SPA, suggesting the absence of residual salivary contamination in the SSH group. Furthermore, FT-IR analysis demonstrated that the spectrum of group SSH was comparable to that of group C and did not include peaks related to saliva components in the range of 1400-1750 cm⁻¹, which were present in the spectra of groups S and SPA. This suggests that salivary contamination was eliminated by cleaning with sodium hydroxide, whereas it partially remained on the sample surface cleaned using phosphoric acid. Therefore, the null hypothesis, which stated that no residual saliva was found on the porcelain surface after cleaning with reagents, was partially rejected.

The SBS of the SPA group was significantly lower than that of group C, although it was significantly higher than that of group S. The failure mode of most of its samples was interfacial failure, indicating that the residual salivary contamination reduced the SBS. In addition, the SBS of group SSH was significantly higher than that of groups S and SPA and similar to that of group C. Moreover, in terms of the failure mode, group SSH showed cohesive failure and mixed failure, similar to group C. These results suggest that cleaning with sodium hydroxide eliminates salivary contamination and restores SBS to a level comparable to that without contamination. Based on these findings, the second null hypothesis, which states that the differences in cleaning agents do not affect the bond strength between the cleaned porcelain and resin cement, was rejected.

The results showed that sodium hydroxide, rather than phosphoric acid, was more effective in cleaning salivary-contaminated porcelain. The present results on the cleaning effect of phosphoric acid are consistent with those of previous studies, which showed that phosphoric acid is less effective in eliminating salivary contamination [25,26]. Contrary to the results of this study, some previous studies have reported that phosphoric acid is effective in eliminating contamination by salivary proteins [22-24]. This contradiction could be related to differences in the grades of phosphoric acid and the cleaning process. Commercially available phosphoric acid used in the past for dental applications had thickening agents like poly (vinyl alcohol) and poly (ethylene oxide) in it, which made it easy to use [32-34]. Thickening agents may remain on the cleaned surface and adversely affect the SBS. In contrast, we prepared reagent grade phosphoric acid without any addition of thickening agents to examine the cleaning effect of phosphoric acid itself. In some previous studies, the contaminated samples were cleaned by directly applying phosphoric acid to the surface of sample from a syringe or by attaching the acid using an applicator. When phosphoric acid is applied using an applicator, salivary contamination can be mechanically eliminated by scratching the surface of the sample. In such cases, the cleaning effect of phosphoric acid on salivary contaminants is affected by the scratching effect.
Sodium hydroxide was more effective than phosphoric acid in eliminating salivary contamination. This suggests that alkalis are more effective in removing salivary contamination of ceramics than acids. This is consistent with the cleaning process in the medical field, where alkalis rather than acids are generally used as cleaning agents to remove protein contamination from surgical instruments. In addition, medical guidelines state that alkali treatment is effective in eliminating protein contamination and restored the SBS to its pre-contamination level. These findings suggest that sodium hydroxide is better than phosphoric acid in eliminating salivary contamination from glass-ceramics.

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Conflicts of interest

The authors declare no conflicts of interest.

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