Original Articles

Different Cleansing Methods Effect to Bond Strength of Contaminated Zirconia

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Abstract

Saliva contamination on a restoration is unavoidable during a try-in procedure. Many studies have shown the negative effect of non-cleansing surface prior to cementation. The aim of this study is to investigate the efficiency of cleansing methods on the shear bond strength of zirconia surfaces. Sixty-six zirconia specimens size 7.5x5.5x0.8 mm were randomly divided into six groups: non-saliva contamination (PC), saliva contamination without surface cleansing (NC), saliva contamination then cleansing with Ivoclean (IC), 0.5 M NaOH solution (NaOH), sandblasting (SB) and sandblasting followed by 0.5 M NaOH solution (SB+NaOH). One specimen in each group was separated for SEM observation. The remaining zirconia specimens were bonded to a composite resin block with Panavia F2.0 and were stored in 37°C distilled water for 24 hours. All specimens were subjected to the SBS test. The data were analyzed by One-way ANOVA and Tukey HSD. The bonded surfaces were observed under stereomicroscope to identify the mode of failure. The results showed that the SBS of saliva contaminated zirconia without surface cleansing (NC) was the significantly lowest (4.62±0.53 MPa) than that of the other groups ($p<0.05$), while SB (14.14±1.72 MPa) and SB+NaOH (15.41±1.65 MPa) were significantly higher than the others ($p<0.05$). However, SB and SB+NaOH showed no statistically significant difference ($p>0.05$). Group PC, IC and NaOH showed no significant difference ($p>0.05$). The mode of failure revealed a greater amount of mixed failure for the majority of SB and SB+NaOH but other groups reveal adhesive failure between zirconia and resin cement for the majority. SEM showed surface morphology changing in SB and SB+NaOH when compared to other group. The saliva contaminated zirconia should be cleaned by Ivoclean, 0.5 M NaOH solution, sandblasting or sandblasting followed by 0.5 M NaOH solution prior to cementation.

Keywords: Bond strength, Saliva contamination, Sodium hydroxide, Surface cleansing, Zirconia

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Introduction

Nowadays, fixed partial restoration is one of the popular choices for restoring a destructed tooth. However, the longevity of the restoration may vary for each person because of many factors including caries, porcelain fracture, dislodgement of crown due to loss of retention, etc. The bonding between the restoration and the tooth structure is one of the most important factors that affect the longevity of the restoration. The bond strength may be affected by contamination on both the tooth and the restoration.

Contamination of saliva during the try-in procedure of restoration is unavoidable. Saliva has a negative effect on bonding which results in shorter longevity but could be solved by using various surface cleansing agents.[1] Many authors suggested the method of cleansing saliva-contaminated surfaces which could be classified into chemical and mechanical cleansing depending on the type of restoration.

The chemical cleansing could be done by using acid etching (i.e. phosphoric acid and hydrofluoric acid) and an alkaline-based agent (Ivoclean). Yang *et al,* 2008 found that phosphoric acid could recover the bond strength of a saliva-contaminated silica-based restoration but at the same time they were found that acid had a negative effect on decreasing the bond strength of a zirconia restoration. Hydrofluoric acid is one choice of surface cleansing of ceramic restoration, both silica-based and zirconia, which could recover bond strength after being saliva-contaminated. The mechanical cleansing could be done by sandblasting and tribochemical on a zirconia restoration. The sandblasting with 50-µm Al_2O_3 at 2.8 bars of pressure for 30 seconds at a distance of 10 mm could improve bond strength of saliva-contaminated zirconia.[2]

Recently, Ivoclean (an alkaline base surface cleansing agent) was introduced to clean the surface of a restoration before the bonding procedure. It desorbed phosphate, saliva phospholipid, from the surface of the restoration. The main composition of Ivoclean is sodium hydroxide (NaOH) solution which results in the improvement of bond strength between the restoration and the tooth structure. Furthermore, the zirconium oxide particles claimed to enhance the phosphate adsorption properties.[2-4] However, comparing the Ivoclean and non-commercial NaOH solution, non-containing zirconium oxide particles, on the shear bond strength of the saliva-contaminated zirconia restoration has not been investigated. The objective of this study is to compare the effect of different surface cleansing methods on shear bond strength of a zirconia restoration.

Materials and Methods

The sixty-six fully sintered zirconia specimens size 7.5x5.5x0.8 mm were prepared from a zirconia block (VITA YZ HT, VITA Zahnfabrik, Germany) and sintered followed the manufacturer's instruction. The specimens were randomly divided into six groups, ten specimens for shear bond strength (SBS) test and another specimen for Scanning electron microscope (SEM) observation (Quanta 250, Thermo Fisher Scientific, USA). The specimens were invested in the center of polyvinyl chloride (PVC) tube with a self-cured acrylic resin (Unifast Trad, GC corporation, Japan) by using a stainless-steel mold.

The 3 mm diameter with 4 mm height composite specimen (Tetric® N-Ceram shade A3.5, Ivoclar Vivadent, Switzerland) was fabricated by an incremental layering technique into the glass covered mold and were light-cured by a LED light curing unit (Elipar™, 3M ESPE, USA) with a light intensity of 1,200 mW/cm^2 for 20 seconds in each increment and an additional 20 seconds on every side before the specimens were removed from the mold. The sixty composite specimens were fabricated and randomly divided into six groups as zirconia specimens. The 0.5 M NaOH solution was prepared and the pH level of the solution was tested by a pH-Fix 0-14 test strip (Macherey-Nagel, Germany) to confirm that the pH level was between 13–13.5. The solution was freshly prepared and kept in a covered volumetric flask.

The surfaces of the specimens were cleaned by immersion in 0.5 M NaOH solution for 8 hours then rinsed with deionized water for 20 seconds and dried with oil free air. The six groups of specimens were surface treated as shown in Figure 1. The sandblasting was operated by Al_2O_3 particles size 50 μm at distance of 10 mm under 0.2 MPa for 20 seconds.

The composite specimens were cemented to the zirconia specimen with Panavia F2.0 (Kuraray Dental, Japan). The cement was mixed by weighting paste A and paste B at about 0.02±0.001 mg using a four-point decimal digital scaler (Sartorius; Germany) as per manufacturer's instructions in mixing ratio of 1:1. The cement was applied on the zirconia surface and the composite specimens were placed under pressure of 1 kg for 8 minutes by a Durometer (ASTM D 2240 Type A, DPTC Instruments, USA). The excessive cement was gently cleaned and lightly cured for 20 seconds on each side following the manufacturer's instructions.

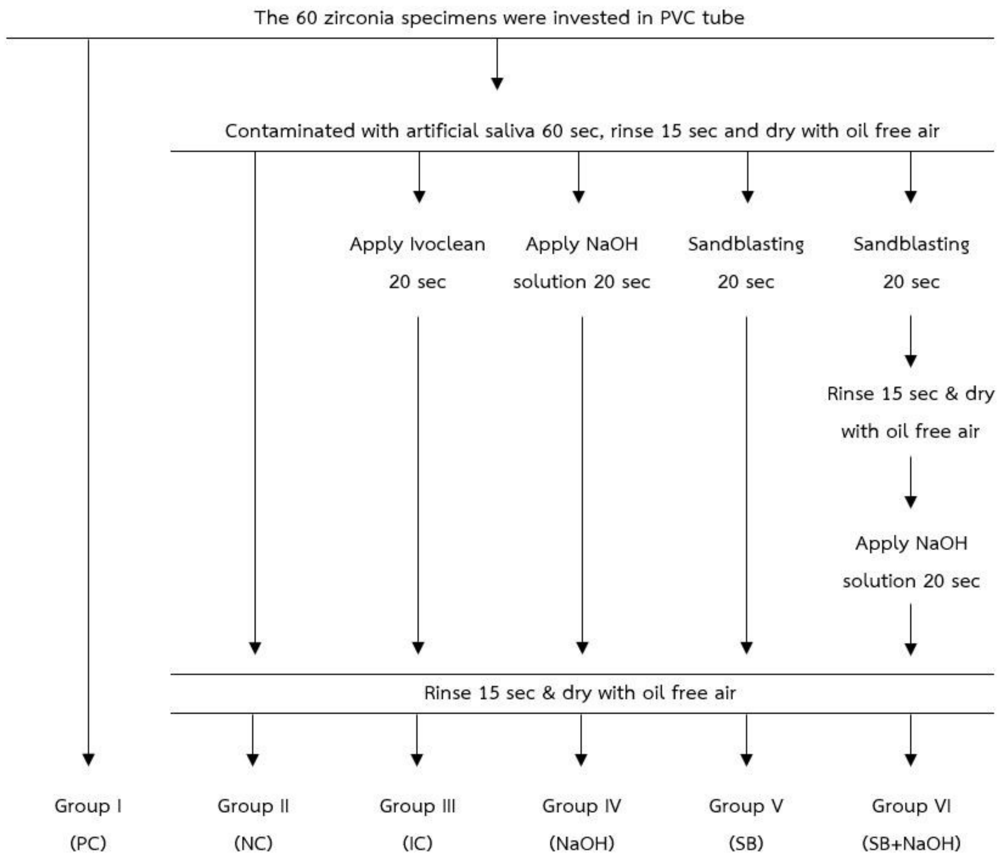

Figure 1 *Methods of surface treated in each group*

Then, the specimens were stored in 37°C distilled water for 24 hours in an incubator (Contherm 1200, New Zealand) before testing the shear bond strength. The shear bond strength test was conducted following the ISO 11405:2015 by Shimadzu universal testing machine (EZ-S, Shimadzu, Japan) with load cell 500 N at cross head speed of 1 mm/min until the bonded surfaces of the specimens were broken. The bonded surfaces of zirconia and the composite resin were investigated under stereomicroscope at 20x magnification to classify the mode of failure as adhesive, cohesive or mixed failure as the following:

Adhesive: The failure occurred between the interface of the zirconia specimen to the resin cement (Zr/P) or the resin cement to the composite specimen (P/Cr).

Cohesive: The failure occurred in the resin cement (P) or the composite specimen (Cr).

Mixed: The failure occurred in a combination of adhesive and cohesive that one of the failure occurred on more than 25 % of the bonded interface.

The six zirconia specimens were surface treated as the above methods. The surface morphologies were observed under SEM at 1000, 3000 and 5000x magnification.

The shear bond strength values were recorded and statistically analysis by One-way Analysis of Variance (ANOVA) and Tukey HSD at 95 % confidence level with SPSS 17 (SPSS Inc, Chicago, Illinois).

Results

Shear bond strength

The mean amount of shear bond strength was analyzed for normal distribution by the Komolgorov-Smirnov test and the data showed normal distribution in each group. Then, the data was analyzed by One-way ANOVA at 95 % confidence level. The results showed that there were significant differences between the groups. Thus, the null hypothesis was not accepted. The multiple comparisons of the shear bond strength were analyzed by Tukey HSD to identify the difference between the groups at 95 % confidence level.

As shown in Table 1, group NC showed the significantly lowest shear bond strength than the experimental groups ($p<0.05$). While group PC, IC and NaOH showed no statistically significant difference ($p>0.05$). Group SB and SB+NaOH showed statistically significant higher difference than the others ($p<0.05$). However, a comparison between group SB and SB+NaOH showed no statistically significant difference ($p>0.05$).

Table 1 Means and standard deviations of the SBS of all of the tested groups in MPa (n = 10)

Group	Mean ± SD (MPa)
PC	6.10 ± 0.62^{B}
NC	4.62 ± 0.53^{C}
IC	6.16 ± 0.62^{B}
NaOH	6.29 ± 0.80^{B}
SB	14.14 ± 1.72^{A}
SB+NaOH	15.41 ± 1.65^{A}

** The same superscript capital letter means there is no significant difference at 95 % confidence level.*

Mode of failure

The fractured interfacial zones on the zirconia and the composite specimens were examined under a stereomicroscope. The major failure of group PC, NC, IC and NaOH is adhesive failure between the zirconia specimens and the resin cement. The difference of group SB and SB+NaOH which had the major failure is mixed type (Table 2).

Table 2 *Mode of failure examined under a stereomicroscope.*

Group	Mode of failure (n)				
	Adhesive		Mixed	Cohesive	
	Zr/P	P/Cr		P	Cr
PC	7	0	3	0	0
NC	9	0	1	0	0
IC	9	0	1	0	0
NaOH	9	0	1	0	0
SB	0	1	9	0	0
SB+NaOH	0	0	10	0	0

Discussion

Surface cleansing methods on shear bond strength of saliva-contaminated zirconia

The number of specimens was calculated by power analysis of the pilot study and the results showed the ten specimens in each group is more than the calculated quantity.

Although the zirconia restoration is fabricated by CAD-CAM technique but the restoration still may not perfectly fit to the abutment and a try-in procedure is still necessary. Saliva contamination is unavoidable in the try-in procedure of the restoration. The saliva contamination on the surface of the zirconia restoration has shown significantly decreasing bond strength in several studies.[1,5] For this reason, the surface of the restoration should be cleaned prior to cementation. In this study, the shear bond strength of group NC has significantly decreased (p<0.05) when compared to a non-saliva contaminated surface (PC) and cleansing the saliva-contaminated surface with different methods; Ivoclean (IC), 0.5 M NaOH solution (NaOH), sandblasting (SB) and sandblasting followed by 0.5 M NaOH solution (SB+NaOH). This finding agreed with previous studies which showed that airborne particle abrasion and cleaning paste yielded higher bond strength value more than the other cleansing methods and airborne particle abrasion yielded the highest bond strength value.[2,5-7]

When the surface of the saliva-contaminated zirconia was cleaned with Ivoclean (IC) and 0.5M NaOH solution (NaOH) for 20 seconds, the shear bond strength was recovered and showed no significant difference to a non-contaminated surface (PC). This result agreed with previous studies that Ivoclean could clean contaminated-zirconia and recover equivalent or a greater bond strength as non-contaminated zirconia.[2,4,6] In this study, the 0.5 M NaOH solution with pH 13–13.5 was investigated due to several studies which found that NaOH solution can desorb phosphate and the amount of desorbed phosphate increased with the increase of the alkalinity of the NaOH solution. In contrast, the lower the pH, the phosphate could better absorb to the zirconia surface.[3,8]

In water pollution management, the zirconia beads are used in phosphate absorption from wastewater. The NaOH solution has been recommended for decontamination of the phosphate-contaminated zirconia before reusing. The phosphate desorption could be done by immersion of the zirconia beads in 0.1 M NaOH for 4 hours and 0.1 M NaOH for 12 hours which show the desorption rate at about 48.0 % and 91.7 % accordingly. The 0.5 M NaOH enhances the desorption of phosphate at more than 0.1 M NaOH but there was no significant difference.[8] However, in this study, immersion in NaOH solution for only 20 seconds could recover the same bond strength as non-contaminated zirconia.

The NaOH solution in this study is prepared to a pH of about 13–13.5 which is the same as Ivoclean's conditions. However, the NaOH solution in this study is in the liquid form which is different from Ivoclean which is available as a gel. Gel is easier to use and could stick to the surface because of it's high viscosity. Thus, it was suggested that Ivoclean be used only 1 to 2 drops per time which is different from the liquid form that must be placed in a container such as beaker or small bowl. In the future, the NaOH solution in liquid form may made into a gel by adding glycerin. Moreover, it may also reduce tissue irritation from accidental contact or respiratory tract irritation from the evaporation of NaOH solution.

Sandblasting with 50 μm aluminum oxide particle at distance of 10 mm, pressure 0.2 MPa, for 20 seconds (SB) and sandblasting followed by the NaOH solution (SB+NaOH) shows a higher shear bond strength of saliva-contaminated zirconia than others. This may be caused from the surface characteristic after sandblasting which create more surface area when compared to a non-sandblasted surface. From the study of He *et al.,* the sandblasted surface of the restoration has more surface roughness than non-sandblasting of about 7.3 times and more than SBS at 1.3 times.[9]

Zirconia is acid-resistant but could be etchable under some specific conditions.[10] In a clinical situation, surface roughening of zirconia might be done by sandblasting which creates micromechanical retention to resin cement.[9] Sandblasting increases the surface area, surface energy, surface wettability and flowing ability of resin cement in to micro-retention areas. When the surface area increases, the oxide layer increases too. This causes more chemical bonding between the oxide layer to the MDP and may lead to higher bond strength. However, sandblasting may create surface flaws, reducing the strength of the restoration and accelerate tetragonal to monoclinic phase transformation.[9,11]

Comparing the sandblasting group (SB) with sandblasting followed by NaOH (SB+NaOH), there was no significant difference with the shear bond strength. The above results showed that sandblasting could remove the contamination on the zirconia surface due to the NaOH solution application after sandblasting could not improve any shear bond strength. The average shear bond strength in this study also showed a range of many previous studies, about 12-20 MPa.[4,12-14] This can conclude that only sandblasting with Al_2O_3 particles size 50 μm at distance of 10 mm, pressure 0.2 MPa for 20 seconds on saliva-contaminated zirconia could recover or yield higher the bond strength compared to non-contaminated zirconia.

Mode of failure

Sandblasting is the best way to improve bond strength and to clean the surface. The mode of failure of group SB and SB+NaOH are mostly mixed while shear bond strength show significant improvement over the others group that are mainly adhesive. This may result from sandblasting that roughens the surface which increases the surface area for retention of the crown by cementation.

The SEM showed that the surface morphology in groups PC, NC, IC, and NaOH were not different. Thus, after surface contamination and cleansing by using NaOH solution or Ivoclean did not affect the surface morphology. However, a more irregular surface was observed in group SB and SB+NaOH compared to the others (Fig. 2). This mode of failure from group SB and SB+NaOH has proven that bond strength is better after being cleaned and surface treated by sandblasting. Therefore, the zirconia restoration should be sandblasted before cementation for better retention.

In further studies, the surface of the zirconia should be investigated by Energy Dispersive Spectrometry (EDS) which can analyze the organic and inorganic elements to detect the amount of phosphate before/after contamination and after cleansing. Zirconia was chosen for investigation of zirconia-oxide which play an important role of chemical bonding to MDP. Thus, the non-precious metal restoration should be further investigated due to the surface of non-precious metal composed of metal-oxide which could bond chemically to MDP in the same way as zirconia.

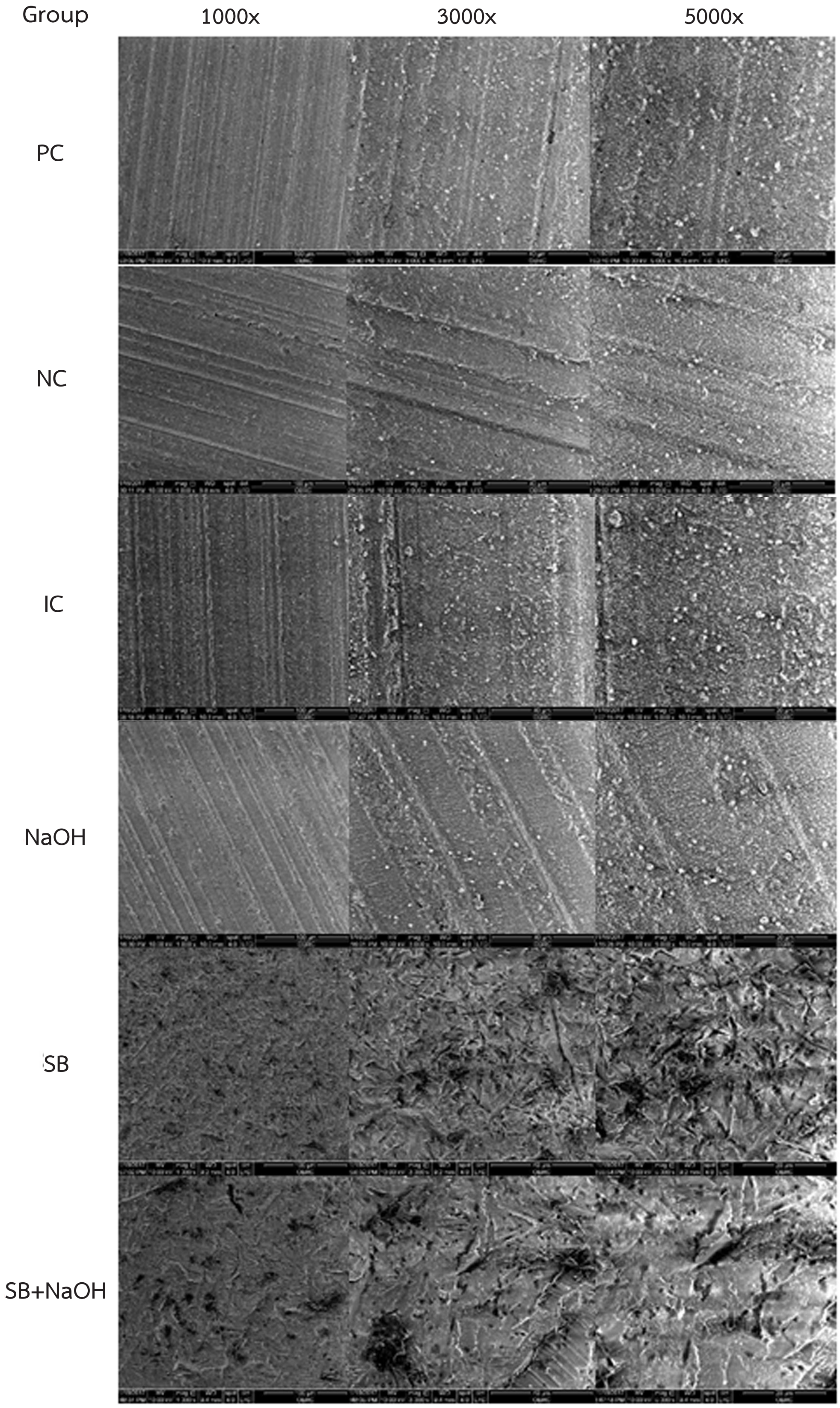

Figure 2 *SEM of zirconia specimens at 1000x, 3000x and 5000x magnificent level*

Conclusion

Saliva contamination significantly decreases the shear bond strength of a zirconia restoration. The recommended effective cleaning methods to remove saliva contamination and recover bond strength are following:

1. Chemical cleansing methods: Application of Ivoclean or 0.5 M NaOH solution for 20 seconds.

2. Mechanical cleansing methods: Sandblasting with Al_2O_3 particles size 50 μm at distance of 10 mm, pressure 0.2 MPa for 20 seconds.

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