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타액으로 오염된 지르코니아 수복물의 접착강도에 세척 방법들이 미치는 영향

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〈Abstract〉

The effect of cleaning methods on bond strength of zirconia after saliva contamination

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본 연구는 MDP 적용 후 타액으로 오염된 지르코니아 수복물을 다양한 방법으로 세척한 후 전단결합강도를 비교하여 세척방법이 결합강도에 미치는 영향을 알아보고자 한다.

80개의 지르코니아 시편을 8개의 군으로 나누었다. 모든 시편에 MDP를 적용한 후 한 개의 군(음성대조군)을 제외하고 나머지 군에 타액을 적용하여 오염시켰다. 그 중 한 개의 군(양성대조군)은 타액 오염 후 세척하지 않고 즉시 레진 시멘트를 이용하여 접착하였다. 나머지 6개의 군의 시편을 물을 이용하여 세척하고 MDP를 적용하거나(물+MDP) 적용하지 않은 군(물), Ivoclean으로 세척하고 MDP를 적용하거나(IVOCLEAN+MDP) 적용하지 않은 군(IVOCLEAN), 차아염소산나트륨을 이용하여 세척하고 MDP를 적용하거나(NaOCl+MDP) 적용하지 않은 군(NaOCl)으로 분류하였다. 모든 시편은 37°C 증류수에 24시간 저장한 후 전단강도를 측정하였고, ANOVA, Tukey's *post hoc* test를 이용하여 전단강도를 분석하였고, MDP의 재적용 여부가 미치는 영향에 대해서는 student *t*-test를 이용하여 통계분석하여 다음의 결과를 얻었다.

양성대조군이 가장 낮은 전단강도 값을 나타냈으며, 물군과 NaOCl군이 낮은 전단강도 값을 나타내며 양성대조군과 유의한 차이가 없었다. IVOCLEAN군은 물군과 NaOCl군보다 유의하게 높은 전단강도 값을 나타내며 음성대조군과 유의한 차이가 없었다. MDP를 재적용한 것은 물과 차아염소산나트륨을 이용한 경우 MDP를 재적용하지 않은 경우와 유의한 차이를 나타내면서 음성대조군과 유의한 차이가 없었다. Ivoclean을 사용한 경우 MDP 재적용 여부와는 관계없이 음성대조군과 유의한 차이가 없었다.

결론적으로, 세척방법에 따라 전단강도는 영향을 받으며, MDP 재적용 여부와는 관계없이 Ivoclean이 효과적이며, 물과 차아염소산나트륨 사용시에는 MDP를 다시 적용해주는 것이 결합강도를 향상시킬 수 있는 방법으로 사료된다.

Key words: Zirconia, Saliva, Cleaning, NaOCl, Ivoclean, Shear bond strength

Ⅰ. INTRODUCTION

Zirconia is a white crystalline oxide of zirconium that has been used in dentistry since the end of the 1990s (Manicone et al., 2007; Vagkopoulou et al., 2009; Thompson et al., 2011; Özcan et al., 2012). Zirconia-based restorations are used widely in dentistry because of their excellent optical and mechanical properties (Manicone et al., 2007;

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Vagkopoulou et al., 2009; Özcan et al., 2012). In addition, the introduction of computer-aided design/computer-aided manufacturing (CAD/CAM) systems has contributed to the increased popularity of zirconia restorations in dentistry (Manicone et al., 2007; Vagkopoulou et al., 2009).

Adhesion between restorations and resin-based cement is a crucial factor that affects the longevity of restorations. When the adhesion is insufficient, the adhesion itself can be weakened by microleakage and secondary caries can occur (Thompson et al., 2011; Özcan et al., 2012).

Silica-based ceramics, which are still used widely, are subjected to phosphoric acid or hydrofluoric acid treatments to roughen their surface and are then treated with silane, which increases their durability by enhancing the mechanical interlocking and chemical bonding with the resin-based cement (Thompson et al., 2011).

Unlike silica-based ceramics, however, phosphoric acid and hydrofluoric acid cannot be used effectively on zirconia. Many zirconia surface treatments, such as airborne-particle abrasion with alumina, selective infiltration etching and tribochemical silica coatings, have been introduced to achieve durable cementation (Thompson et al., 2011). Kern and Wagner reported that the application of resin-based cement containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) resulted in high bond strength (Kern et al., 1998). Since then, many researchers have focused on MDP-containing resin cement or bonding/silane coupling agents (Gargari et al., 2010; Thompson et al., 2011). Many studies reported that the application of a MDP primer improved the bond strength of resin-based cement to zirconia due to chemical bonding between the MDP and zirconia (Koizumi et al., 2012; de Souza et al., 2014; Yi et al., 2015; Pilo et al., 2016). Primers containing MDP have been used widely in zirconia cementation procedures.

During the try-in procedure, the zirconia surface is contaminated by saliva. Some studies reported that saliva contamination affects the bond strength and durability of resin bonding to zirconia by reducing the adaptation of the restoration materials to the bonded surface (Yang et al., 2008; Phark et al., 2009; Aladağ et al., 2014; Feitosa et al., 2015; Ishii et al., 2015; Kim et al., 2015; Angkasith et al., 2016; Tunc et al., 2016).

Phosphoric acid is effective in removing organic contaminants, which led to various experiments aimed at applying phosphoric acid to remove saliva contamination from the zirconia surface. On the other hand, the results showed that the cleaning procedure with phosphoric acid weakened the bond strength. The residual phosphorous from phosphoric acid bonded with zirconia, which hindered bonding between MDP and zirconia and had a negative effect on adhesion (Yang et al., 2008; Phark et al., 2009; Feitosa et al., 2015; Ishii et al., 2015; Angkasith et al., 2016; Tunc et al., 2016). Previous studies showed that air-abrasion appears to be an effective cleaning method to remove saliva contaminants but many studies suggested that additional air-abrasion may affect the zirconia phase transformation and weaken the zirconia ceramic adhesion (Yang et al., 2008; Phark et al., 2009; Chintapalli et al., 2013; Feitosa et al., 2015; Ishii et al., 2015; Tunc et al., 2016).

Recently, a commercial universal cleaning solution (Ivoclean; Ivoclar Vivadent, Schaan, Lichtenstein) was launched. The manufacturer claimed that this cleaning solution could be used effectively in ceramic and metal restorations.

In dental clinics, sodium hypochlorite (NaOCl) is used widely and is very effective in removing organic materials (Dikmen et al. 2015). One study reported that NaOCl was effective in cleaning zirconia surfaces (Kim et al., 2015). On the other hand, another study reported that the effectiveness of NaOCl in cleaning zirconia was uncertain (Aladağ et al., 2014).

Therefore, this study evaluated the effects of various cleaning methods on the shear bond strength of zirconia to resin cement after saliva contamination.

II. MATERIALS AND METHODS

1. Specimen Preparation

Eighty zirconia disk specimens (Zirmon; Kuwotech Co., Ltd., Gwangju, Korea), 15 mm in diameter and 1 mm in thickness, were prepared. All the specimens were wet-grounded with 320 and 600 grit silicon carbide paper for 5 minutes. After wet-grinding, the samples were air-abraded with 50 μm Al_2O_3 for 10 seconds at 3 bar at a distance of 10mm. All specimens were cleaned ultrasonically in 95% ethanol for 10 minutes, rinsed with water, and air-dried. The specimens were divided into 8 groups (n=10). All groups were first treated with one coat of MDP primer (Z-prime Plus; Bisco, Schaumburg, IL, USA).

The saliva used in study was collected from a non-smoking man 2 hours after eating and drinking. This study was approved by the Institutional Review Board of Pusan National University Dental Hospital (IRB, PNUDH-2016-040). NaOCl was prepared by diluting a NaOCl solution (Fresh Rox; Malguennara, Busan, Korea) to a concentration of 5 % to 1 % by mixing with deionized water. The negative control (group 8) was bonded with resin cement after the MDP coating and without saliva contamination. All specimens (except the negative control, group 8) were contaminated with human saliva by rubbing a microbrush on the zirconia surface for 20 seconds. The positive control (group 7) was not cleaned after contamination. With the exception of groups 7 and 8, they were cleaned by rinsing with water for 20 seconds and air-dried for 15 seconds (groups 1 and 4), or cleaned with a cleansing solution (Ivoclean) according to the manufacturer's instructions (groups 2 and 5, agitated for 20 seconds using a microbrush, rinsed with water for 10 seconds and air-dried for 15 seconds), or cleaned with 1% NaOCl (agitated for 20 seconds with a microbrush) and rinsed with water for 10 seconds and air-dried for 15 seconds (groups 3 and 6). Groups 4 to 6 were treated with one coat of MDP primer again after the cleaning procedure.

All bonding procedures were carried out immediately after the contamination and cleaning procedures.

2. Bonding Procedure

Resin blocks were made with a tube, 2.5 mm in diameter and 1 mm in thickness, filled with composite (Filtek Z100;

Table 1. Materials used in this study

Material	Brand	Composition	Manufacturer
Zirconia	Zirmon	94.7% ZrO_2, 5.2% Y_2O_3	Kuwotech Co., Ltd., Gwangju, Korea
Primer	Z-PRIME Plus	MDP, BPDM, methacrylates, ethanol	Bisco, Schaumburg, IL, USA
Composite	Filtek Z100	BisGMA, TEGDMA	3M ESPE, St Paul, MN, USA
Resin Cement	RelyX U200	**Base:** methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives **Catalyst:** methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	3M ESPE, St Paul, MN, USA
Clean paste	Ivoclean	Zirconium oxide, Water, Polyethylene glycol, Sodium hydroxide, Pigments, additives	Ivoclar Vivadent Schaan, Lichtenstein
	Fresh Rox	Sodium hypochlorite, Water	Malguennara, Busan, Korea

Abbreviations; MDP: 10-methacryloyloxydecyl dihydrogen phosphate, BPDM: biphenyl dimethacrylate; BisGMA: bisphenol-A-diglycidylether dimethacrylate, TEGDMA: triethylene glycol dimethacrylate

3M ESPE, St Paul, MN, USA). After the specimens received the cleaning procedure, resin cement (Rely X U200; 3M ESPE, St Paul, MN, USA), which was mixed according to the manufacturer's instructions, was placed on the zirconia surface and a resin block was positioned on resin cement and photopolymerized for 10 seconds using a LED curing unit (SmartLite PS; Dentsply DeTrey, Konstanz, Germany) at 1000mW/cm^2. All specimens were stored in distilled water at 37°C for 24 hours.

3. Shear Bond Strength

The shear bond strength was measured using a universal testing machine (Electromechanical 3345 All-electric Test Instrument; Instron Industrial Products, Grove City, PA, USA) at a crosshead speed of 1 mm/min after water storage. The load was applied to the adhesive interface until failure. The maximum stress was obtained when failure occurred.

4. Statistical analysis

The data was analyzed statistically by an analysis of variance (ANOVA) and Tukey's *post hoc* test at the $p<0.05$ level. A Student's *t*-test was used to compare the shear bond strength according to the re-coating of MDP after the cleaning procedure (SPSS 20.0; SPSS Inc., Chicago, IL, USA).

Ⅲ. RESULTS

Tables 2 and 3 list the results of the shear bond strength test. The results showed significant differences in shear bond strength among the groups. Group 7 (positive control) showed the lowest bond strength. Groups 1 and 3 showed low shear bond strength and did not show significant difference with group 7. Group 2 showed significantly higher shear bond strength than groups 1 and 3, and did not show significant difference with the group 8 (negative control). A comparison of the single coating of MDP and re-coating of MDP showed a significant difference between groups 1 and 4 and between groups 3 and 6 but there was no significant difference between groups 2 and 5.

Table 2. Mean shear bond strength values and standard deviations (SD) of the cleaning and control groups (MPa)

Group	Mean±SD
Group 1: MDP, saliva, water	14.62±1.28[a]
Group 2: MDP, saliva, Ivoclean	17.94±1.17[b]
Group 3: MDP, saliva, NaOCl	15.49±1.56[a]
Group 7: MDP, saliva (posivive control)	13.72±3.79[a]
Group 8: MDP (negative control)	18.81±1.59[b]

Comparisons are valid only for same parameter.
Values are mean ± SD, n=10 (per group). Different superscript letters indicate statistically significantly different groups ($p<0.05$).
Abbreviation; MDP: 10-methacryloyloxydecyl dihydrogen phosphate

Table 3. Mean shear bond strength values and standard deviations of MDP coat (once) and re-coat (twice) (MPa)

	Cleaning Method		
Priming conditions	Water	Ivoclean	NaOCl
MDP one coat	Group 1 : 14.62±1.28	Group 2 : 17.94±1.17	Group 3 : 15.49±1.56
MDP re-coat	Group 4 : 18.17±1.38	Group 5 : 19.39±1.29	Group 6 : 17.32±1.28
t-tset	$P<0.05$	$P>0.05$	$P<0.05$

Values are mean ± SD, n=10 (per group).
Abbreviation; MDP: 10-methacryloyloxydecyl dihydrogen phosphate

Ⅳ. DISCUSSION

This study examined the effect of the cleaning methods on the shear bond strength of self-adhesive resin cement to saliva-contaminated zirconia surface. Saliva contains organic materials, such as saliva protein, food debris, bacteria, and enzyme molecules, as well as inorganic materials in a water solution (Yang et al; 2008). Prior to cementation, contamination by saliva inside the restoration is unavoidable and removal of this contamination is crucial for the long-term durability of the restoration. Organic contaminants, including saliva proteins, are difficult to remove, and if they remain, they have negative effects on adhesion by altering the surface state of zirconia (Yang et al., 2008; Phark et al., 2009; Aladağ et al., 2014; Feitosa et al., 2015; Ishii et al., 2015; Kim et al., 2015; Angkasith et al., 2016; Tunc et al., 2016). The results of this study confirmed that the saliva contamination had a negative effect on the resin cement and zirconia surface.

Previous studies showed that water rinsing was not effective in removing saliva contamination from the zirconia surface zirconia (Yang et al., 2008; Phark et al., 2009; Feitosa et al., 2015; Ishii et al., 2015; Tunc et al., 2016). On the other hand, Angkasith et al. reported that after the application of MDP primer prior to salivary contamination, water rinsing is effective in recovering the bond strength (Angkasith et al., 2016). The results of the present study showed that water rinsing is ineffective in removing saliva contamination even if MDP primer is applied prior to saliva contamination. Although the zirconia surface becomes more hydrophobic after applying MDP, the adhesion of saliva to the surface could not be avoided due to its amphiphilic property (de Souza et al., 2014; Hanning, 1997). In a previous study, XPS analysis revealed the existence of the remaining carbon residue even after cleaning with water, suggesting that contaminants, such as saliva protein, cannot be removed completely with water once they adhere to the surface of the restoration material (Yang et al., 2008; Phark et al).

A previous study showed that NaOCl is effective in removing saliva contamination from the zirconia surface (Kim et al., 2015). In this study, cleaning with NaOCl was not effective in removing saliva contamination from the zirconia surface. The results showed that the zirconia surface cleaned with NaOCl has no significantly different adhesion strength compared with the zirconia surface cleaned with water. Although NaOCl is effective in dissolving organic material and proteins, it has been reported that in cases where MDP was applied prior to saliva contamination, the cleaning process by microbrush could remove the MDP coating, resulting in decreased adhesion (Tunc et al., 2016). Moreover, when NaOCl remained on the surface, it hindered polymerization of the adhesive, and decreased the bond strength of the resin cement (Dikmen et al., 2015).

Recently, a new commercial cleaning solution called Ivoclean was launched on the market. Ivoclean is an alkaline suspension of zirconium dioxide particles in water that absorbs phosphate contaminants like a sponge in the media, leaving a clean surface. In this study, the Ivoclean group showed a higher bond strength than the other cleaning methods. According to the manufacturer's instructions, MDP should be re-coated after cleaning with Ivoclean and then rinsed with water (Ivoclean Product Brochure 2011). However, re-coating of MDP did not show significant difference with single coating of MDP. Therefore, Ivoclean maintained the bond strength regardless of the MDP re-coating.

After the cleaning procedure, a re-coating of MDP produced a higher bond strength, and recovered the bond strength to near its original value. As mentioned previously, MDP is removed partially during the cleaning procedures (Tunc et al., 2016). Therefore, the chemical bond recovered through re-coating with MDP. Moreover, re-coating of MDP

will enhance the bond strength by increasing the surface wettability, which was lowered by contact with saliva or moisture (de Souza et al., 2014; Yi et al., 2015; Ishii et al., 2015).

There are no reports on the application of MDP both before and after the cleaning procedure. Re-coating of MDP in clinical examples may be an effective method for recovering the shear bond strength.

One limitation of this study was that the aging values were not taken. Therefore, further study will be needed to compare the cleaning methods under aging conditions.

V. CONCLUSIONS

Within the limitation of this study, water rinsing and NaOCl were found to be ineffective in removal saliva contaminants from the zirconia surface. On the other hand, Ivoclean was found to be effective regardless of the re-coating of MDP. After cleaning, re-coating MDP resulted in a higher shear bond strength than the case where water or NaOCl was used without a MDP re-coating. Therefore, when water or NaOCl is used to remove surface contaminants, a re-coating of MDP has a positive effect on cementation.

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ABSTRACT

The effect of cleaning methods on bond strength of zirconia after saliva contamination

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This study evaluated the effects of various cleaning methods on the shear bond strength of zirconia ceramics after saliva contamination. Eighty zirconia disk specimens were divided into 8 groups. All groups were treated with one coat of MDP primer. All specimens (except the negative control) were contaminated with human saliva on the zirconia surface. The positive control went through the bonding procedure immediately after contamination without any cleaning procedure. With the exception of control groups, the remaining six groups were rinsed with water and either applied with MDP recoating (WATER+MDP) or without MDP recoating (WATER). While some were cleaned with a Ivoclean with MDP recoating (IVOCLEAN+MDP) or not applied with MDP recoating(IVOCLEAN), others were cleaned with a 1% NaOCl solution with MDP recoating (NaOCl+MDP) or without MDP recoating (NaOCl). The shear bond strength of all specimens were measured after being stored in distilled water at 37°C for 24 hours. The data was analyzed statistically by an analysis of ANOVA, Tukey's post hoc test and Student's t-test was used to compare the shear bond strength according to the re-coating of MDP after the cleaning procedure. The positive control group showed the lowest shear bond strength value, and the WATER group and NaOCl group showed no significant difference when compared to the positive control group. The IVOCLEAN group showed significantly higher shear bond strength when compared to Water group and NaOCl group but not with the group of negative control. After rinsing with water or the NaOCl solution, the comparison of the single coating of MDP and re-coating of MDP showed different shear bond strengths but there was no significant difference to the negative control. After rinsing with Ivoclean, there was no significant difference to the negative control regardless of the recoating of MDP. In conclusion, the shear bond strength was affected by the cleansing procedure and Ivoclean was found to be effective regardless of the re-coating of MDP. When water or the NaOCl solution is used to remove surface contaminants, the re-coating of MDP provides a positive effect on cementation.

Key Words: Zirconia, Saliva, Cleaning, NaOCl, Ivoclean, Shear bond strength