Efficacy of various cleaning solutions on saliva-contaminated zirconia for improved resin bonding

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PURPOSE. This study aimed to investigate the efficacy of cleaning solutions on saliva-contaminated zirconia in comparison to air-abrasion in terms of resin bonding. MATERIALS AND METHODS. For saliva-contaminated air-abraded zirconia, seven cleaning methods—no contamination (NC), water-spray rinsing (WS), additional air-abrasion (AA), and cleaning with four solutions (Ivoclean [IC]; 1.0 wt% sodium dodecyl sulfate [SDS], 1.0 wt% hydrogen peroxide [HP], and 1.0 wt% sodium hypochlorite [SHC])—were tested. The zirconia surfaces for each group were characterized using various analytical techniques. Three bonded resin (Panavia F 2.0) cylinders (bonding area: 4.5 mm²) were made on one zirconia disk specimen using the Ultradent jig method [four disks (12 cylinders/group); a total of 28 disks]. After 5,000 thermocycling, all specimens were subjected to a shear bond strength test with a crosshead speed of 1.0 mm/minute. The fractured surfaces were observed using an optical and scanning electron microscope (SEM). RESULTS. Contact angle measurements showed that groups NC, AA, IC, and SHC had hydrophilic surfaces. The X-ray photoelectron spectroscopy (XPS) analysis showed similar elemental distributions between group AA and groups IC and SHC. Groups IC and SHC showed statistically similar bond strengths to groups NC and AA (P>.05), but not groups SDS and HP (P<.05). For groups WS, SDS, and HP, blister-like bubble formations were observed on the surfaces under SEM. CONCLUSION. Within the limitations of this in vitro study, some of the cleaning solutions (IC or SHC) were effective in removing saliva contamination and enhancing the resin bond strength. [J Adv Prosthodont 2015;7:85-92]

KEY WORDS: Zirconia; Saliva; Cleaning agent; Dental bonding

INTRODUCTION

Recently, yttria partially stabilized tetragonal zirconia (Y-TZP) has come into wide clinical use mainly due to its high fracture strength.1,2 Although zirconia ceramic restorations may be luted using conventional luting cements,2 bonding with resin to the ceramic would be advantageous for many clinical applications.3 Wegner and Kern3 demonstrated that a durable bond to zirconia was achieved by applying resin luting cements containing 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) to an air-abraded zirconia surface.

Resin-ceramic bonding might be compromised in clinical situations when compared with clean laboratory situations.1 After the try-in of all-ceramic restoration, the ceramic surface might be contaminated by saliva, blood, or sili-
cone fit-indicators. Among them, saliva contamination is reportedly the main cause of decreased resin bond strength. Some previous studies demonstrated that saliva contamination significantly affected the strength and durability of resin bonds to zirconia and that air-abrasion was the most useful cleaning method.

Recently, a commercial cleaning solution (Ivoclean, Ivoclar Vivadent, Schaan, Liechtenstein) has been introduced to the dental market. The manufacturer claims that a simple application of the solution, followed by water rinsing and air-drying, effectively cleans the saliva-contaminated bonding surfaces of various dental restorations including zirconia ceramic. However, little research has been carried out with respect to the cleaning efficacy of such cleaning solutions on saliva-contaminated zirconia in terms of resin-zirconia bonding.

In this in vitro study, we tested the cleaning efficacy of four (one commercial and three experimental) cleaning solutions in enhancing resin-zirconia bonding following simulation of try-in with saliva exposure and compared it to that of air-abrasion. The hypothesis tested was that the cleaning solutions are less effective than air-abrasion in removing saliva contaminants from zirconia surfaces with respect to zirconia bonding with a 10-MDP-containing resin cement.

**MATERIALS AND METHODS**

Saliva was collected from one non-smoking male who had refrained from eating and drinking 1.5 hours before the collection procedure, in accordance with the Institutional Review Board of Kyungpook National University Hospital (BMRI 74005-452) and with the informed consent of the donor. All experiments were performed with fresh saliva. Ivoclean (IC, lot #: R78201), which contains zirconium oxide, water, polyethylene glycol, sodium hydroxide, pigments, and additives, was tested. Sodium dodecyl sulfate (SDS), hydrogen peroxide (HP, H₂O₂), and sodium hypochlorite (SHC, NaOCl) were purchased from Bio-Rad (Richmond, CA, USA; lot #: L1610301) or Duksan Pure Chemicals (Seoul, Korea; lot #: IC8EB41 and 032516, respectively) and diluted with distilled water into 1.0 wt% solutions. The solution codes were likewise used for designating each group in this study.

Zirconia (Lava, 3M ESPE, Seefeld, Germany) disk specimens (20 mm diameter and 1.5 mm thickness) were fabricated according to the manufacturer’s instructions. Initially, one surface of all specimens was polished with 600 grit silicon carbide (SiC) paper, air-abraded with 50 μm Al₂O₃ at 0.25 MPa for 15 seconds at a distance of 10 mm, ultrasonically cleaned in isopropyl alcohol for 3 minutes, rinsed with water, and finally air-dried. The specimens were classified into seven study groups. Except for the control group (group NC, no saliva contamination), all specimens were immersed in saliva for 1 minute and rinsed with water-spray for 15 seconds and air-dried for another 15 seconds. In group WS, no further cleaning was performed. In group AA, specimens were air-abraded, ultrasonicated, rinsed, and air-dried as described above. In group IC, a microbrush was used to apply IC, which was allowed to react for 20 seconds, followed by rinsing with water-spraying for 15 seconds and air-drying for another 15 seconds, according to the same procedure as above.

**Fig. 1.** Design of this study. All specimens were water-rinsed and finally air-dried prior to further procedures.
the manufacturer's recommendation. In groups SDS, HP, and SHC, the corresponding solutions were applied, respectively, rinsed, and air-dried in the same way as for group IC. The study design for surface analysis and shear bond strength testing is illustrated in Fig. 1.

To assess the cleanliness of the surfaces for each study group, water contact angle (CA) measurements were performed. Since roughness may also alter the CA values, surface roughness measurements were performed prior to CA analysis.3,9,10 The surface roughness R\textsubscript{s} of each specimen was measured using a profilometer (Surftest SFV-400, Mitutoyo Corp., Kawasaki, Japan) at a stylus speed of 0.1 mm/second, a cutoff of 0.8 mm, and a range of 600 μm. The R\textsubscript{s} of each specimen was determined as the average of five readings (n=5/group). The CA measurements were performed using a CA goniometer (OCA 15 plus, Data Physics Instrument GmbH, Filderstadt, Germany) in a temperature-controlled room at 23 ± 1°C with relative humidity at 50 ± 5%.11 Using the dynamic sessile drop method, the advancing CA of water was measured after settling 6 μL droplets on the material surface, the receding one then being measured after sucking 2 μL from the droplet into the syringe (n=5/group).2,9,12,13 The CA hysteresis (H) was calculated using the equation: \( H = \cos \Theta_a - \cos \Theta_r \), in which \( \Theta_r \) and \( \Theta_a \) are the receding and advancing water CAs, respectively.14 The degree of correlation between the cos\( \Theta \) and the H was determined by the Pearson correlation coefficient.

To determine the effectiveness of the cleaning methods, specimens of the seven test groups were examined with X-ray photoelectron spectroscopy (XPS).1,5,6 All measurements were performed using an XPS system (PHI Quantera SXM, ULVAC-PHI Inc., Tokyo, Japan) with an X-ray source providing Al Ka X-rays and kinetic energy of 1486.6 eV.1 The emission angle of the photoelectrons was kept constant at 45°. A 180° hemispherical analyzer with 32 channel detectors was used for detection of the photoelectrons. A wide scan survey spectrum (0-1100 eV) was obtained to examine the surface composition of the specimens under ultra high vacuum at 10\textsuperscript{-7} Pa.7 High resolution scans of the carbon (C1s), oxygen (O1s), zirconium (Zr3d), nitrogen (N1s), and aluminum (Al2p) peaks were obtained.1,5 Ratios of C/O, C/Zr, O/Zr, N/Zr, and Al/Zr were calculated.

For shear bond strength testing, a total of 28 zirconia disks were prepared and embedded in round silicone rubber molds using an acrylic resin. The uncovered (to be bonded) surfaces were treated according to the study design (Fig. 1) and isolated using a bonding jig (Ultradent Products Inc., South Jordan, UT, USA).5,9,15 Freshly-mixed Panavia F2.0 (Kuraray Noritake Dental Inc., Okayama, Japan; lot #: 00586D (A paste), 00114D (B paste, light shade)) was applied to the surface by packing the material into cylindrical-shaped plastic matrices with an internal diameter of 2.38 mm and then irradiated for 20 seconds using a halogen curing light (Elipar TriLight, 3M ESPE; output intensity=750 mW/cm\textsuperscript{2}).9 In this manner, three bonded resin cylin-
derers were made on one zirconia disk specimen and a total of 12 resin cylinders (i.e., four disk specimens) prepared for each group. Prior to debonding, all bonded specimens were stored in water at 37°C for 24 hours and then thermocycled 5000 times between 3°C and 55°C water baths with a dwell time of 30 seconds and a transfer time of 5 seconds between each bath.16

The specimens were perpendicularly engaged at their bonded resin cylinder bases with a round-notched custom shear blade in a universal testing machine (Model 3343, Instron Inc., Canton, MA, USA) at a crosshead speed of 1.0 mm/minute until bonding failure occurred.3,15 Bond strengths (MPa) were calculated from the peak load of failure (N) divided by the bonded surface area. Following debonding, all fractured interfaces were examined under an optical microscope (SMZ800, Nikon Corp., Tokyo, Japan) at 10× magnification to determine the failure mode: A, adhesive failure at the zirconia-resin interface; C, cohesive failure within resin; and M, a combination of these failure modes (mixed failure). In addition, a scanning electron microscope (SEM, JSM-6700F, Jeol, Japan) operating at 5 kV was used to observe the debonded zirconia surfaces.

The Shapiro-Wilk normality test and Levene’s variance homogeneity test were applied to the surface roughness and bond strength data. The surface roughness data, which met both the normality and variance homogeneity assumptions, were analyzed using one-way ANOVA. As the bond strength data were normally distributed but showed inhomogeneity of variances between groups, they underwent a log\textsubscript{10} transformation to meet homogeneity of variance prior to analysis (Leven’s test, P=135).14 Shear bond strength comparisons between the seven test groups were conducted using one-way ANOVA followed by the Bonferroni post hoc test. The analyses were done under an assumption of independence among the three resin cylinders bonded to each zirconia disk specimen (ST 1).17 In addition, a simple random effect in mixed model ANOVA was conducted to allow correlation between the resin cylinders (ST 2).17 Statistical analyses were carried out using SPSS 17.0 for Windows (SPSS Inc., Chicago, IL, USA). Differences were considered statistically significant at P≤.05 (marginally significant at P≤.1, highly significant at P≤.01, and extremely significant at P<.001).18 In addition, a post hoc power analysis was carried out to examine the power of the bond strength data using G\textsuperscript{*}Power 3.1.7 software.

RESULTS

Table 1 summarizes the surface roughness values of the zirconia specimens. One-way ANOVA revealed no significant differences among the seven groups tested (P=.683), indicating that the additional air-abrasion cleaning (group AA) did not significantly increase the R\textsubscript{s} value of the primary air-abrasion (group NC).

Fig. 2 shows the results of the Pearson correlation analysis between the cosine values of the advancing CAs (cos\( \Theta \)) and the contact angle hysteresis (H). Groups NC,
AA, IC, and SHC showed higher $\cos \Theta_a$ values, whereas groups WS, SDS, and HP yielded lower values. A significant strong negative correlation was found between the parameters ($r = -0.866, P = 0.012$).

The results of the XPS analysis are shown in Fig. 3 and Table 2. The peak intensity ratios of C/O and C/Zr were the highest in group WS (2.1 and 11.6, respectively). The ratios were reduced after air-abrasion (group AA), being comparable to those of group NC. The four cleaning solution groups showed C/O and C/Zr ratios that were similar to group AA—except for group SDS, which exhibited notably higher ratios. The N element was not detected only in groups NC and AA. In group WS and HP, the phosphorus (P) element was also detected (0.5 and 0.9 at%, respectively). In group SDS, 0.7 at% sulfur (S) element was also detected. On the other hand, group SHC showed 0.5 at% chlorine (Cl).

Table 1. Ra surface roughness (μm) of the zirconia specimens (mean ± SD, n=5)

| Groups     | $R_a$       |
|------------|-------------|
| NC (control) | 0.16 ± 0.02 |
| WS (water-spray) | 0.17 ± 0.02 |
| AA (air-abrasion) | 0.17 ± 0.02 |
| IC (Ivoclean) | 0.17 ± 0.01 |
| SDS (sodium dodecyl sulfate) | 0.17 ± 0.02 |
| HP ($H_2O_2$) | 0.17 ± 0.03 |
| SHC (NaOCl) | 0.16 ± 0.01 |

There were no significant differences among the test groups (one-way ANOVA, $P = 0.683$).

Fig. 2. Pearson’s correlations between the $\cos \Theta_a$ and the contact angle hysteresis ($H$). $r$ indicates the Pearson’s correlation coefficient. AA: air-abrasion; HP: $H_2O_2$; IC: Ivoclean; NC: control; SDS: sodium dodecyl sulfate; SHC: NaOCl; WS: water-spray.

Table 2. Ratios of carbon (C), oxygen (O), nitrogen (N), and aluminum (Al) elements

| Groups           | C/O   | C/Zr  | O/Zr  | N/Zr  | Al/Zr |
|------------------|-------|-------|-------|-------|-------|
| NC (control)     | 0.5   | 2.7   | 5.4   | -     | 1.2   |
| WS (water-spray) | 2.1   | 11.6  | 5.6   | 0.7   | 1.1   |
| AA (air-abrasion)| 0.6   | 3.4   | 5.9   | -     | 1.4   |
| IC (Ivoclean)    | 0.6   | 3.1   | 5.0   | 0.2   | 1.0   |
| SDS (sodium dodecyl sulfate) | 0.9   | 5.6   | 5.9   | 0.3   | 1.3   |
| HP ($H_2O_2$)    | 0.6   | 3.5   | 5.7   | 0.2   | 1.2   |
| SHC (NaOCl)      | 0.7   | 3.2   | 4.8   | 0.2   | 0.9   |

Fig. 3. Wide-scan XPS spectra for the test groups. AA: air-abrasion; HP: $H_2O_2$; IC: Ivoclean; NC: control; SDS: sodium dodecyl sulfate; SHC: NaOCl; WS: water-spray. Al: aluminum; C: carbon; Cl: chlorine; O: oxygen; P: phosphorus; S: sulfur; Zr: zirconium. The peaks between 600 and 0 eV are shown.
Table 3 summarizes the shear bond strength and failure mode results. Each *P* value from post hoc comparison Bonferroni’s test is presented in Table 4. Group SHC showed the highest shear bond strength value (10.9 ± 1.7 MPa). Groups NC, AA, IC exhibited statistically similar bond strength values to that of group SHC. In contrast, groups SDS, WS, and HP showed significantly lower bond strengths than the aforementioned four groups (i.e., groups SHC, NC, AA, and IC). For groups SHC, NC, AA, and IC, mixed failures outnumbered adhesive failures. For groups SDS and HP, the higher frequency of adhesive failures observed when compared to mixed failures. For group WS, all failures were adhesive.

**Table 3.** Shear bond strength (MPa) of the test groups and type of failure mode

| Groups               | Shear bond strength | Failure modes |
|----------------------|---------------------|---------------|
|                      | Mean ± SD           | A             | M             |
| SHC (NaOCl)          | 10.9 ± 1.7*a        | 4†            | 8             |
| NC (control)         | 10.4 ± 2.1a         | 5             | 7             |
| AA (air-abrasion)    | 9.5 ± 2.4a          | 6             | 6             |
| IC (Ivoclean)        | 9.1 ± 1.3a          | 5             | 7             |
| SDS (sodium dodecyl sulfate) | 6.7 ± 1.6b       | 8             | 4             |
| WS (water-spray)     | 5.7 ± 1.2bc         | 12            | 0             |
| HP (H2O2)            | 4.4 ± 0.8c          | 10            | 2             |

*a* Means with same lowercase letters are not statistically different at *P* >.05. The *P* values are shown in Table 4.

† Number of resin cylinders. A: adhesive failure at the zirconia-resin interface; M: a combination of adhesive failure at the interface and cohesive failure within resin.

**Table 4.** The *P* values of the shear bond strength data from post hoc comparison Bonferroni’s test

| Statistics | Groups | WS   | AA   | IC   | SDS  | HP   | SHC |
|------------|--------|------|------|------|------|------|-----|
| ST 1       | NC     | <.001| >.999| >.999| <.001| <.001| >.999|
|            | WS     | <.001| <.001| >.999| .098 | <.001|
|            | AA     | >.999| .002 | <.001| >.999|
|            | IC     | .004 | <.001| <.001| .760 |
|            | SDS    | <.001| <.001| <.001|
|            | HP     | <.001|      |      |      |
| ST 2       | NC     | <.001| >.999| >.999| .002 | <.001| >.999|
|            | WS     | <.001| .001 | >.999| .331 | <.001|
|            | AA     | >.999| .030 | <.001| >.999|
|            | IC     | .043 | <.001| >.999|
|            | SDS    | .007 | <.001|      |
|            | HP     | <.001|      |      |

ST 1: the analyses were done under the assumption of independence among the three resin cylinders bonded to each zirconia disk specimen. ST 2: a simple random effect in mixed model ANOVA was conducted to allow correlation between the resin cylinders. Means were log10 (MPa) transformed prior to analysis. AA: air-abrasion; HP: H2O2; IC: Ivoclean; NC: control; SDS: sodium dodecyl sulfate; SHC: NaOCl; WS: water-spray.

**DISCUSSION**

According to the manufacturer, IC contains sodium hydroxide and is meant for extraoral use only. The three substances used to prepare the cleaning solutions also have potentially adverse intraoral effects when present in high concentrations and with prolonged exposure. Clinically relevant concentrations of SDS are 0.015-1.5%, and toothpastes usually contain 1-3% of SDS as the detergent. Home mouth rinses and dentifrices contain low concentrations (1% or less) of HP. Although 5.25% SHC is a common tissue solvent, 1% SHC solution has effective tissue-dissolving capability. Considering the potential use of such cleaning solutions for intraoral repair procedures with restorative composite resin, three experimental zirconia-cleaning solutions with relatively low concentration (1.0 wt%) were prepared and tested (Fig. 1).

Non-covalent adsorption of salivary proteins, simulated by saliva immersion in this study, occurs on zirconia surface after try-in. Only water-spray rinsing of the specimens after saliva contamination significantly lowered the bond strength value, compared to the control (Table 3). The additional air-abrasion after contamination effectively removed saliva contamination without significantly increasing the *R* value (Table 2) and restored the bond strength significantly to the same level as that in the control, in accordance with some previous studies. In this study, moreover, some of the cleaning solutions (IC or SHC) were...
found to be also effective in removing saliva contamination and in enhancing the resin bond strengths. Thus, the null hypothesis that the cleaning solutions are less effective than air-abrasion in terms of resin bond strength was rejected.

Surface hydrophobicity/hydrophilicity can be determined by CA measurement. It is known that surface roughness also alters the CA values of the surface. In this study, the influence of surface roughness on the CA values can be excluded because the $R_a$ values among the seven test groups were not significantly different (Table 1). Although zirconia is rather hydrophobic and has a low surface free energy, air-abrasion creates high surface energy and promotes micoretention. Thus, air-abraded zirconia surfaces without saliva contamination may be considered relatively hydrophilic. According to the CA measurements (Fig. 2), groups NC, AA, IC, and SHC, which produced higher bond strength values (Table 3 and Table 4), showed lower advancing CA values (39.7-52.2°) and therefore indicated more hydrophilic surfaces. On the contrary, groups WS, SDS, and HP, which exhibited lower bond strength values, yielded lower CA values (62.9-65.2°) and indicated more hydrophobic surfaces. Thus, more hydrophilic zirconia surfaces indicate more effective cleaning; whereas more hydrophobic surfaces indicate less-effective cleaning. In addition, since chemical heterogeneity can also cause CA hysteresis, greater surface inhomogeneity due to less-effective cleaning of saliva-contaminated zirconia surfaces may induce greater CA hysteresis. Thus, a significantly strong negative correlation between the $\cos \Theta_a$ and the CA hysteresis (Fig. 2) indicates that CA hysteresis is also relevant in assessing the cleanliness of rough surfaces. Nonetheless, CA measurements alone are not sufficient to characterize surface chemical changes on zirconia before and after cleaning. Therefore, XPS was also used to identify the chemical elements on the surfaces. The depth and spatial resolutions for XPS are 1-25 nm and 8-150 μm, respectively. Since the subtended zirconia surface signal was detectable (Fig. 3), the thickness of the contamination layer was less than 10 nm.

In dental practice, SHC solution has been widely used as an endodontic irrigant due to its effective antimicrobial and tissue-dissolving capabilities. It is also known that residual SHC may interfere with resin polymerization due to oxygen generation. Among the experimental cleaning solutions, however, 1% SHC solution was the most effective in removing the saliva contaminants from the zirconia surface (Table 3). XPS analysis also showed a lower O/Zr ratio for the zirconia surface cleaned with SHC than that cleaned with HP. SEM observation of the debonded surface revealed little bubble formation at the interface (Fig. 4G). These findings may indicate that SHC effectively cleaned the surface; water-spray rinsing then removed most of the residual SHC on the zirconia surface.

The results for group AA confirms again that air-abrasion is a useful cleaning method of saliva-contaminated zirconia. In clinical practice, however, the more complex surface geometry of zirconia-based restorations may make it difficult to remove contamination using air-abrasion. In such cases, a microbrush would be more convenient for applying cleaning solutions such as SHC to the inner surfaces of the restorations.

According to the manufacturer (Ivoclar Vivadent Scientific Documentation, 2011), the alkaline suspension of zirconium oxide particles in IC removes salivary phosphate con-
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In this study, a zirconia disk, which is expected to show less heterogeneity as opposed to tooth material, was used. This means whether the cleaning solutions tested are also effective for such contamination. The cleaning efficacy of 1% SHC solution on other prosthetic restoration surfaces should also be studied. In addition, the safety of SHC should be assessed for intraoral use.

CONCLUSION

The findings of this study confirm that saliva contamination significantly reduces resin shear bond strength to zirconia and that air-abrasion is a useful cleaning method. However, a simple application of IC or 1% SHC effectively removed the saliva contaminants and provided a clean surface. The resin bond strength results were supported by water CA measurements and chemical identification of the zirconia surface with XPS. However, long-term clinical studies are still required to clarify the efficacy of the cleaning solutions in improving resin bonding of saliva-contaminated zirconia.

REFERENCES

1. Yang B, Scharnberg M, Wolfart S, Quaas AC, Ludwig K, Adelung R, Kern M. Influence of contamination on bonding to zirconia ceramic. J Biomed Mater Res B Appl Biomater 2007;81:283-90.
2. Kim MJ, Kim YK, Kim KH, Kwon TY. Shear bond strengths of various luting cements to zirconia ceramic: surface chemical aspects. J Dent 2011;39:795-803.
3. Wegner SM, Kern M. Long-term resin bond strength to zirconia ceramic. J Adhes Dent 2000;2:139-47.
4. Quaas AC, Yang B, Kern M. Panavia F 2.0 bonding to contaminated zirconia ceramic after different cleaning procedures. Dent Mater 2007;23:506-12.
5. Yang B, Lange-Jansen HC, Scharnberg M, Wolfart S, Ludwig K, Adelung R, Kern M. Influence of saliva contamination on zirconia ceramic bonding. Dent Mater 2008;24:508-13.
6. Yang B, Wolfart S, Scharnberg M, Ludwig K, Adelung R, Kern M. Influence of contamination on zirconia ceramic bonding. J Dent Res 2007;86:749-53.
7. Phark JH, Duarte S Jr, Kahn H, Blatz MB, Sadan A. Influence of contamination and cleaning on bond strength to modified zirconia. Dent Mater 2009;25:1541-50.
8. Boulange-Petermann L, Joud JC, Baroux B. Wettability parameters controlling the surface cleanability of stainless steel. In: Mittal KL, ed. Contact angle, wettability and adhesion. Leiden; VSP; 2008. p. 139-51.
9. Kim YK, Son JS, Kim KH, Kwon TY. Influence of surface energy parameters of dental self-adhesive resin cements on bond strength to dentin. J Adhesion Sci Technol 2013;27:1778-89.
10. Kim YK, Min BK, Son JS, Kim KH, Kwon TY. Influence of different drying methods on microtensile bond strength of self-adhesive resin cements to dentin. Acta Odontol Scand 2014;72:954-62.
11. Takimoto M, Ishii R, Iino M, Shimizu Y, Tsujimoto A, Takamizawa T, Ando S, Miyazaki M. Influence of temporary cement contamination on the surface free energy and dentine bond strength of self-adhesive cements. J Dent 2012;40:131-8.
12. Chibowski E, Hołysz L, Terpiłowski K, Jurak M. Investigation of super-hydrophobic effect of PMMA layers with different fillers deposited on glass support. Colloids Surf A: Physicochem Eng Asp 2006;291:181-90.
13. Hołysz L, Miroslaw M, Terpiłowski K, Szczęś A. Influence of relative humidity on the wettability of silicon wafer surfaces. Ann UMCS Chem 2008;63:223-39.
14. Phark JH1, Duarte S Jr, Blatz M, Sadan A. An in vitro evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface. J Prosthodont Dent 2009;101:29-38.
15. Hagge MS, Lindemuth JS. Shear bond strength of an autopolymerizing core buildup composite bonded to dentin with 9 dentin adhesive systems. J Prosthodont Dent 2001;86:620-3.
16. Yun JY, Ha SR, Lee JB, Kim SH. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. Dent Mater 2010;26:650-8.
17. Sattabanasuk V, Vachiramon V, Qian F, Armstrong SR. Resin-dentin bond strength as related to different surface preparation methods. J Dent 2007;35:467-75.
18. McGee MD, Rinhart RD, Ngo I., Eisen EA, Kelsey KT, Wiencke JK, Herrick RF. Urinary 1-hydroxypyrene and polycyclic aromatic hydrocarbon exposure among asphalt paving workers. Ann Occup Hyg 2004;48:565-78.
19. Neppelberg E, Costea DE, Vintermyr OK, Johansson AC. Dual effects of sodium lauryl sulphate on human oral epithelial structure. Exp Dermatol 2007;16:574-9.
20. Walsh IJ. Safety issues relating to the use of hydrogen perox-