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RESEARCH AND EDUCATION

Influence of different airborne-particle abrasion pressures, cleaning methods, and artificial aging on zirconia ceramic bonding

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ABSTRACT

Statement of problem. Airborne-particle abrasion (APA) with alumina combined with a primer or luting resin containing a phosphate monomer has been well established for resin bonding to zirconia. However, a standardized method for bonding to zirconia is unavailable, as some factors including the optimal pressure of airborne-particle abrasion, the cleaning method, and the luting resin system are still unclear because of the limited available data.

Purpose. The purpose of this in vitro study was to evaluate the influence of different airborne-particle abrasion pressures and different cleaning methods of zirconia on the tensile bond strength (TBS) of 2 luting resins system containing methylacryloyloxydecyl-dihydrogenphosphate (MDP) either in the primer or in the luting resin.

Material and methods. A total of 128 disk specimens were milled from zirconia blocks and divided into groups according to the pressure of airborne-particle abrasion of either 0.1 MPa or 0.25 MPa (n=64). The specimens were then divided into 2 subgroups and were either cleaned in an ultrasonic device in isopropanol or with oil-free air stream (n=32). The subgroups were further divided according to the luting resin (n=16) and the bonding surfaces were perpendicularly luted to the acrylic resin tubes filled with a composite resin (Clearfil F II) with either an MDP-containing primer and a luting resin or with a self-adhesive MDP-containing luting resin. Each of the 8 groups was further divided into 2 subgroups (n=8) with either 3–days water storage at 37 °C or 150–days water storage with additional thermal cycling (37 500 thermal cycles between 5 °C and 55 °C) for artificial aging. The TBS was evaluated with a universal testing machine. All specimens underwent failure mode analysis. Statistical analysis was performed with 4-way ANOVA and, if required, with 3-way ANOVA (α=.05).

Results. The mean ±standard deviations ranged from 57.0 ±11.1 MPa to 61.5 ±7.1 MPa after 3 days and from 32.3 ±11.7 MPa to 61.0 ±12.4 MPa after 150 days. The pressure of airborne-particle abrasion and the cleaning method had no significant effect on the TBS (P>.05), whereas the luting resin and artificial aging significantly influenced the TBS (P<.05).

Conclusions. Irrespective of the pressure of airborne-particle abrasion and the cleaning method, the use of an MDP-containing luting resin for bonding to zirconia was found to be more advantageous compared with an MDP-containing primer and luting resin because of higher bond strength and no significant decrease after artificial aging. (J Prosthet Dent 2025;134:230-236)

To use the advantages of zirconia ceramics and non-retentive minimally invasive restorations a long-term durable resin bonding is of clinical importance. In vitro bonding studies and a few long-term clinical studies have shown different methods on pretreating, roughening, and activating the zirconia surface before bonding.[1–5] Pretreatments such as simply cleaning, polishing, or grinding, and omitting airborne-particle abrasion (APA) on zirconia ceramic surfaces have been reported to not provide reliable bond strengths regardless the primer, whereas APA with alumina combined with a primer or luting resin containing a phosphate monomer, methylacryloyloxydecyl-dihydrogenphosphate (MDP), has shown favorable outcomes with durable long-term bonding to zirconia ceramic.[6–14]

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Clinical Implications

The findings of this study are promising regarding the use of the MDP-containing luting resin Panavia SA Cement Universal for bonding to zirconia, because of higher bond strength and no significant decrease after artificial aging, compared with the luting resin Panavia V5 with its special MDP-containing primer. The pressure of airborne-particle abrasion on zirconia at 0.1 MPa or 0.25 MPa did not affect the TBS. As the use of 99% isopropanol in an ultrasonic device as a cleaning method did not increase the TBS, cleaning with oil-free air stream after APA might be a time saving method in clinical practice.

APA produces an activated microroughened zirconia surface, increases the bonding surface, and modifies the surface energy and wettability, and enhances the formation of resin-ceramic micromechanical interlocking.[6,7] However, adequate APA on zirconia is challenging, as no surface alteration is visible to the naked eye. To ensure adequate APA of the bonding surface with no areas omitted, coloring of the bonding surface before APA has been advocated.[15] The applied pressure of APA of studies bonding to zirconia ceramic has ranged from 0.05 MPa to 0.4 MPa.[9,10,16–20] However, the effect of APA pressure on the bond strength appears controversial with studies recommending a lower pressure of 0.05 MPa or 0.1 MPa[9,18,19] and studies recommending a higher pressure of 0.2 MPa or 0.25 MPa for a durable bond strength.[11,16]

After APA, alumina particles might be retained on the bonding surface which might influence the long-term bond durability.[21] Therefore, different cleaning methods have been suggested. However, a standardized method of cleaning zirconia before bonding is unavailable. Ultrasonic cleaning of the zirconia surfaces in 70%[9] or 99%[18,22] isopropanol, in 96%[10,17] or 99%[11,16] ethanol, in deionized[16,19] or distilled[23] water has been described. Additionally, hydrofluoric acid, tap water,[19] or simply cleaning with oil-free air stream have been tested for cleaning zirconia before bonding.[13,23,24] Nevertheless, the effect of ultrasonic cleaning is still controversial and the research data are limited. Cleaning zirconia with oil-free air stream has been reported to result in higher bond strength values compared with cleaning ultrasonically,[23] whereas other studies showed higher bond strength values for ultrasonically cleaned than for oil-free air stream cleaned zirconia.[13,24]

Owing to the sensitivity of the bonding technique[25,26] manufacturers have developed universal primers[13] and universal self-adhesive luting resins that contain MDP monomers and silane coupling agents that can be applied to all restorative surfaces, eliminating the prerequisite of a separate primer and providing a one-step bonding technique.[12,27] MDP-containing primers or luting resins have been reported to increase the bond strength to airborne-particle abraded zirconia unlike conventional primers or luting resins without MDP.[7–9,16,24,28–30] MDP activates the chemical interactions of the hydroxyl groups in the zirconia surface with the ester group of the MDP monomer, leading to durable and strong chemical bonding.[1,2] Despite simplifying the bonding procedure, the effectiveness of self-adhesive luting resins with different components and their possible chemical interactions incorporated into a single composition and the comparison of bond strength to a luting resin and a separate MDP-containing primer has not been thoroughly addressed. However, a few studies have shown promising results for such an MDP-containing self-adhesive luting resins without the need for a separate primer.[31–33]

Therefore, the aim of this in vitro study was to evaluate the durability of tensile bond strength (TBS) of 2 different luting resin systems, containing MDP either in the primer or in the luting resin, on zirconia ceramic after different methods of airborne-particle abrasion and cleaning. The null hypotheses were that the pressure of airborne-particle abrasion, the cleaning method of the airborne-particle abraded zirconia surface, the luting resin system, and artificial aging would have no significant effect on the TBS.

MATERIAL AND METHODS

One hundred and twenty-eight disk specimens (Ø 8×3.5 mm) were milled out of 3Y-TPZ zirconia (ICE Zirkonia; ZirkonZahn). The bonding surfaces of all specimens were wet polished with 600-grit abrasive silicon carbide paper (CarbiMet; Buehler) in a grinding machine (Ecomet250 pro; Buehler).

A total of 128 acrylic resin tubes (17-mm length, 3.2-mm inner diameter) were filled with an autopolymerizing composite resin (Clearfil F II; Kuraray Noritake Dental Inc), which was allowed to polymerize for 7 minutes. During the polymerizing, the specimens were pretreated according to the protocol of the respective group.

The specimens were divided into 2 groups (n=64), color-marked (Schneider Maxx 240; Schneider Schreibgeräte GmbH), and airborne-particle abraded at a 10-mm distance with 50–µm Al_2O_3 (Orbis Dental) at a pressure of either 0.1 MPa (1) or 0.25 MPa (25) (Spot blasting unit P-G 400; Harnisch+Rieth GmbH+Co KG) until the marked color had been removed. The specimens of each group were then divided into 2 subgroups (n=32) according to the cleaning methods after APA and were either cleaned in 99% isopropanol (Otto Fischar

GmbH Co KG) in an ultrasonic device (Ultrasonic Finevo Cleaning System; bredent group GmbH Co KG) for 3 minutes with the surface to be bonded facing downwards, dried with oil-free air stream (U), followed by allowing to dry for a further 3 minutes or the specimens were cleaned with oil-free air stream (A) for 15 seconds (Spot blasting unit P-G 400) only. Each subgroup (1-U, 1-A, 25-U, and 25-A) was further divided into 2 subgroups (n=16) according to the luting resin, using Panavia V5 (PV) or Panavia SA Cement Universal (PS) (Kuraray Noritake Dental Inc). For groups luted with Panavia V5 (A2 Kuraray Noritake Dental Inc) the bonding surface of specimens was conditioned with its special primer (Clearfil Ceramic Primer Plus; Kuraray Noritake Dental Inc) and dried with oil- and water-free air spray. The acrylic resin tubes were luted perpendicularly to the bonding surfaces of the specimens with the luting resin of the respective group in a bonding apparatus with a load of 7.4 N. The excess luting resin was gently removed by foam pellets (Schaumstoff-Pellets Ø 4 mm; Pluradent AG), followed by applying an oxygen inhibitor (Oxygard II; Kuraray Noritake Dental Inc) and initially polymerizing (radii-cal LED curing light; SDI) from 2 sides for 20 seconds 2 minutes after the beginning of mixing the luting resin. Subsequently, the specimens were then further polymerized for 180 seconds (HiLite power; Kulzer GmbH). The bonded specimens were stored in an incubator (INCU-Line; VWR International LCC) in 37 °C water. Each of the 8 subgroups (1-U-PV, 1-A-PV, 25-U-PV, 25-A-PV, 1-U-PS, 1-A-PS, 25-U-PS, and 25-A-PS) was finally divided into 2 subgroups (n=8) according to the storage protocol. The specimens were either stored in 37 °C water for 3 days (3) or were stored for 150 days (150) and were subjected to thermocycling (THE-1100; SD Mechatronik) with 37 500 thermal cycles (7500 per month), a temperature of 5 °C and 55 °C, and a dwell time of 30 seconds. After the allocated storage period, the bond strength of the luting resin to the differently pretreated zirconia surfaces was evaluated with a TBS test in a universal testing machine using a chain loop alignment (Zwick Z010; ZwickRoell) at a crosshead speed of 2 mm/minutes (Fig. 1).

The fractured interfaces of all debonded specimens were examined visually with a light microscope (Wild M420 Heerbrugg AG) at ×25 magnification to assess the failure mode. The failure mode was classified as a cohesive failure in the luting resin or the composite resin and as an adhesive failure at zirconia surface. Representative images of failure modes were made with a scanning electron microscope (SEM) (Zeiss Supra 55 V; Zeiss) after sputtering a 10-nm gold alloy layer (Leica EM QSG 100; Leica Microsystems GmbH).

The data were evaluated with a statistical software program (IBM SPSS Statistics, v20.0 for Windows; IBM Corp) (α=.05 for all tests). The Shapiro Wilk test was performed, which revealed that the data were normally distributed with the exception of the group 1-A-PV-3D (*P*=.011). The homogeneity of the variances was tested with the Levene test, which was not significant. Statistical analysis was performed with 4-way ANOVA and, if required, with 3-way ANOVA. Four-way ANOVA revealed no 4-way interaction or any 3-way interaction. However, 2-way interaction between luting resin and aging (*P*<.001) was revealed.

Figure 1. Bonded specimen in universal testing machine.

RESULTS

The 4-way ANOVA results are reported in Table 1. The mean ±standard deviation values of the TBS are shown in Table 2 and ranged from 57.0 ±11.1 MPa to 61.5 ±7.1 MPa after 3 days and from 32.3 ±11.7 MPa to 61.0 ±12.4 MPa after 150 days.

The APA pressure (*P*=.429) and the cleaning method (*P*=.898) had no significant influence on the TBS. However, the luting resin used significantly affected the TBS. Groups with the luting resin Panavia V5 (1-U-PV, 25-U-PV, 1-A-PV, and 25-A-PV) showed significantly lower TBS than groups with the luting resin Panavia SA Cement Universal (1-U-PS, 25-U-PS, 1-A-PS, and 25-A-PS) after 150 days (*P*<.001). Artificial aging significantly affected the TBS with groups of the luting resin Panavia V5 (1-U-PV, 25-U-PV, 1-A-PV, and 25-A-PV) showing significantly lower TBS than groups of the luting resin Panavia SA Cement Universal (1-U-PS, 25-U-PS, 1-A-PS, and 25-A-PS) after 150 days (*P*<.001).

The prevalence of failure modes of test groups is shown in Figure 2. The failure mode was predominantly cohesive for all groups. Higher percentages of adhesive failure were observed for groups of the luting resin

Table 1. Test of between-subjects' effects (4-way ANOVA)

Source	Type III Sum of Squares	Df	Mean Square	F	*P*
Corrected Model	14260.297[a]	15	950.686	9.677	<.001
Intercept	361683.630	1	361683.630	3681.695	<.001
Luting resin	3961.390	1	3961.390	40.324	<.001
Pressure	61.827	1	61.827	.629	.429
Cleaning method	1.616	1	1.616	.016	.898
Aging	5733.331	1	5733.331	58.361	<.001
Luting resin × pressure	152.077	1	152.077	1.548	.216
Luting resin × cleaning method	134.193	1	134.193	1.366	.245
Luting resin × aging	3755.194	1	3755.194	38.225	<.001
Pressure × cleaning method	1.213	1	1.213	.012	.912
Pressure × aging	143.863	1	143.863	1.464	.229
Cleaning method × aging	25.956	1	25.956	.264	.608
Luting resin × pressure × cleaning method	2.327	1	2.327	.024	.878
Luting resin × pressure × aging	8.789	1	8.789	.089	.765
Luting resin × cleaning method × aging	89.111	1	89.111	.907	.343
Pressure × cleaning method × aging	20.130	1	20.130	.205	.652
Luting resin × pressurex cleaning method × aging	169.280	1	169.280	1.723	.192
Error	11002.695	112	98.238		
Total	386946.622	128			
Corrected total	25262.992	127			

Dependent variable: tensile bond strength.

[a] R Squared=0.564 (adjusted R Squared=0.506)

Table 2. Tensile bond strength (TBS) with mean ±standard deviation (SD) values (in MPa) for all groups

Group	TBS (MPa)	
	3 Days Mean ±SD	150 Days Mean ±SD
1-U-PV	60.9 ±6.1[aA]	35.5 ±13.0[aB]
25-U-PV	57.0 ±11.1[aA]	32.4 ±6.4[aB]
1-A-PV	59.4 ±8.1[aA]	41.7 ±11.0[aB]
25-A-PV	61.5 ±7.1[aA]	32.3 ±11.7[aB]
1-U-PS	57.9 ±7.3[aA]	61.0 ±12.4[bA]
25-U-PS	61.3 ±8.0[aA]	58.3 ±11.1[bA]
1-A-PS	59.7 ±10.8[aA]	54.7 ±10.0[bA]
25-A-PS	61.0 ±10.7[aA]	55.8 ±10.0[bA]

Different superscript lowercase letters indicate significant differences within each column for different luting resin with same pressure of airborne-particle abrasion, cleaning method and storage protocol (*P*≤.05).

Different superscript uppercase letters indicate significant differences within each row for different storage protocols in groups with same pressure of airborne-particle abrasion, cleaning method, and luting resin (*P*≤.05).

Panavia V5 after 150 days storage (1-U-PV, 25-U-PV, 1-A-PV, and 25-A-PV). Representative SEM images of failure modes are shown in Figure 3.

DISCUSSION

Based on the results of this study the null hypothesis that the pressure of airborne-particle abrasion would have no significant effect on the TBS was not rejected, as the tested APA pressures did not statistically influence the TBS. This finding was consistent with another study that reported no statistically significant differences between the APA pressures of 0.1 MPa and 0.25 MPa on zirconia when ultrasonic cleaning in 99% isopropanol, and luting with Panavia SA Cement Plus or Multilink Hybrid Abutment (Ivoclar AG) was performed.[18] However, this study evaluated the retention of zirconia copings. A more comparable study tested the TBS of an MDP-containing luting resin (Panavia 21; Noritake Dental Inc) with APA pressure of either 0.05 MPa or 0.25 MPa and different cleaning methods and also reported high, durable resin bonding with high- and low-pressure APA, which did not differ significantly.[19] This study reported a mean TBS of 42.8 MPa after 3 days and 36.3 MPa after 30 days with thermocycling (7500 cycles) for APA pressure of 0.25 MPa and ultrasonic cleaning in 99% isopropanol, which was lower than the groups 25-U-PV (57 MPa) and 25-U-PS (61.3 MPa) of the current study after 3 days and also lower than group PS-25-U (58.3 MPa) after 150 days storage time. As the methodology was consistent with the present study, the differences in the TBS could be explained by the use of different luting resins. Differences of bond strength between luting resins of the same manufacturer have been observed previously.[32] Two other studies, however, were also in agreement with the findings of the present study reporting no significant differences in TBS for different pressures.[10,17]

An influence of the APA pressure on the TBS has been reported in other studies.[9,16] Significant higher TBS was reported for 0.05 MPa and 0.1 MPa than for 0.25 MPa, but for the use of an MDP-free luting resin system.[9] In contrast, a pressure of 0.2 MPa, other than 0.1 MPa or 0.4 MPa, has been reported to provide durable and reliable bonding.[16] This could be explained by the effect of ultrasonic cleaning in deionized water and ethanol or possible contamination.

In the present study, however, no significant difference was obtained for different cleaning methods after APA. Therefore, the null hypothesis that the cleaning method of the airborne-particle abraded zirconia surface would have no significant effect on the TBS was also not rejected. These findings were not in agreement with the

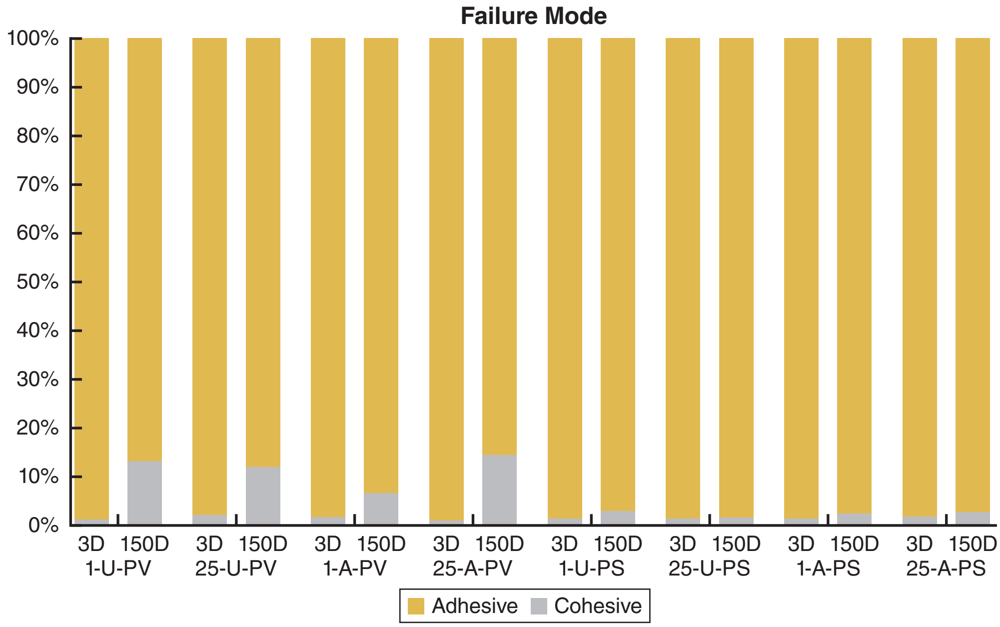

Figure 2. Bar chart of prevalence of failure modes (in percent) of all groups after tensile bond strength testing.

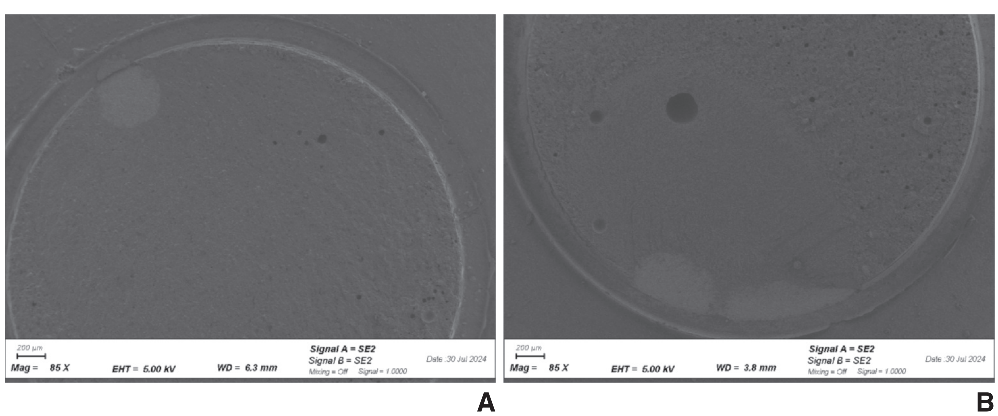

Figure 3. Representative scanning electron microscope images. A, Predominantly cohesive failure mode (25-A-PS). B, Mixed failure mode (1-A-PV). Original magnification ×85.

outcomes of 2 studies which investigated the same 2 cleaning methods as the current study.[13,24] Both studies reported a significant decrease of TBS for the groups that were airborne-particle abraded and cleaned with compressed air after either 30 days[24] or 150 days[13] compared with the groups that were ultrasonically cleaned in 99% isopropanol after APA. The obtained TBS of 33 MPa after 150 days for the group with ultrasonic cleaning[13] was comparable with the ultrasonically cleaned groups 1-U-PV (35.5 MPa) and 25-U-PV (32.4 MPa) of the current study, but not with the groups 1-U-PS and 25-U-PS. This might be because of the similarity of using an MDP-containing primer. However, the different outcome of these could also be explained by the use of different luting resins (Multilink Automix; Ivoclar AG) but also by the different grain size (110 µm) for APA. Another study controversially reported a negative effect of ultrasonic cleaning on bonding to silica-coated zirconia.[23] But it can be concluded that using distilled water for ultrasonic cleaning might has affected the highly reactive silica-coated surface other than the ultrasonic cleaning itself.

The null hypothesis that the luting resin would not significantly influence the TBS was rejected, as groups with the luting resin Panavia V5 showed significantly lower TBS in comparison with groups with Panavia SA Cement Universal after 150 days. This finding was consistent with another study that investigated the microtensile bond strength of the same 2 luting resins on zirconia, showing significantly lower values for Panavia V5 than for Panavia SA Cement Universal after thermocycling for 50 000 cycles.[32] These findings, however, contradict the results of another study which reported no differences between TBS for Panavia V5 and Panavia SA Cement Plus Automix (Kuraray Noritake Dental Inc), a previous version of the MDP-containing luting resin, after 150 days.[16] Another study using the same luting resins as in the current study reported statistically comparable TBS after 3 and 150 days, but also showed lower TBS for Panavia V5 (13.6 MPa) than for Panavia SA Cement Universal (24.7 MPa), and notably lower TBS compared with the current study.[31] Despite using the same study protocol and same aging, the difference in TBS might be attributable to the sensitive and complex technique of adhesive bonding.[25,26] Whereas Panavia SA Cement Universal appeared to be the less sensitive and elaborate procedure because of eliminating the prerequisite of a separate primer compared with the luting resin Panavia V5 and its special MDP-containing primer. Furthermore, different operators might be another explanation for the differences of the obtained TBS, as the influence of the operator on bonding has been shown previously.[34]

The null hypothesis that artificial aging would have no significant effect on the TBS was rejected for all groups luted with Panavia V5 with a significant decrease in TBS after artificial aging, but not rejected for all groups luted with Panavia SA Cement Universal. This result partially agrees with the result of another study, in which the group of Panavia V5 significantly decreased after 150 days regardless the APA pressure of 0.1 or 0.25 MPa. In the contrary, the MDP-containing luting resin only showed a significant decrease at 0.25 MPa, but not at 0.1 MPa.[33] However, Panavia SA Cement Plus was used in the aforementioned study and the results are therefore only comparable to a limited extent. In the current study there was no significant decrease for 1-U-PS and 25-U-PS after artificial aging, but TBS were also lower for 25-U-PS than for 1-U-PS. The negative effect of artificial aging on the bond strength of luting resins has been reported in other studies.[12,31] The decrease in TBS after artificial aging could be explained by deterioration and hydrolytic degradation of the bonding interface caused by water storage and thermal stress.[35] The positive effect of the self-adhesive luting resin Panavia SA Cement Universal after artificial aging, however, could be explained by the less sensitive and elaborate procedure.

The mode of failure after 3–day and 150–day storage was predominantly cohesive in most of the test groups, but with higher percentages of adhesive failure for groups of the luting resin Panavia V5 after 150–days storage. Thus, aging appeared to have an impact on the MDP-containing primer and the luting resin Panavia V5 and its failure mode. This result was consistent with previous studies.[31,33]

With a clinical view of the knowledge gained from these findings, it can be concluded that the TBS of all test groups were acceptable after artificial aging and were notably higher than the reported minimum of clinically acceptable bond strength range of 10 MPa to 13 MPa.[36] However, the MDP-containing luting resin Panavia SA Cement Universal may be preferred to the MDP-containing primer and the luting resin Panavia V5 because of the higher TBS after artificial aging and the less sensitive and elaborate technique. These findings were supported by previous studies in which an MDP-containing luting resin without the need for a separate primer led to higher bond strengths than a luting resin with an MDP-containing primer.[31–33] Significant differences were not found between the APA pressure and cleaning methods, thus, cleaning with air stream after APA might be a promising time saving method in clinical practice.

Limitations of the current study included a relatively low number of specimens in each group and the in vitro experimental design, which did not accurately simulate clinical conditions. The application of thermocycling with no mechanical fatigue during the artificial aging process might be considered as another limitation to the study. Further research including controlled clinical studies are still required to evaluate the durability and strength of the obtained bond strength to the pretreatments of zirconia and the adhesive system.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The pressure of airborne-particle abrasion on zirconia at 0.1 MPa or 0.25 MPa did not affect the TBS of the luting resin.
2. The use of isopropanol in an ultrasonic device as a cleaning method of airborne-particle abraded zirconia surfaces before bonding did not increase the TBS compared with cleaning with oil-free air stream only.
3. The use of Panavia SA Cement Universal appeared to be beneficial because of its higher TBS after artificial aging compared with Panavia V5.
4. Contrary to the luting resin Panavia SA Cement Universal TBS decreased after artificial aging for groups luted with Panavia V5.

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Matthias Kern: Conceptualization, Methodology, Project administration, Supervision, Writing-review and editing. **Frank Lehmann**: Investigation, Resources. **Lisa Türp**: Writing- original draft, Writing- review and editing. **Sebastian Wille**: Formal analysis, Writing-review and editing.

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