# Influence of artificial aging on the shear bond strength of zirconiacomposite interfaces after pretreatment with new 10-MDP adhesive systems

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# Abstract

Introduction: This in-vitro study investigates the bond strength of different zirconia composites with three different modern adhesive systems after artificial aging using thermocycling and water storage. Methods: A total of 90 specimens of zirconia (InCoris, Sirona, Germany, Bernsheim) were ground using a 165 µm grit rotating diamond disc. Thirty specimens were additionally treated with either Futurabond U "FBU" (VOCO GmbH), or Futurabond M+ "FBM" (VOCO GmbH) or Futurabond M+ in combination with the DCA activator "FBMD" (VOCO GmbH). One of the three different types of composites - BifixSE "BS", BifixQM "BQ" or GrandioSO "G" (VOCO GmbH) - was bonded to the ten specimens of each group. All of the specimens underwent artificial aging using thermocycling between 5°C and 55°C for 5000 cycles followed by water storage for 100 days. Shear bond strength (SBS) was determined in a universal testing machine. The type of failure was evaluated using fluorescence microscopy. The data were compared to existing data without artificial aging. Statistical analysis was performed with ANOVA and the Tukev test. Results: FBM and FBMD had higher SBS than FBU in combination with all tested composites, except BifixSE. In nearly all groups, artificial aging had no effect, with the exception of the combination of FBMD with BifixSE, in which there was a significant decrease in SBS after the aging process (p<0.001). Conclusion: The new 10-MDPcontaining adhesive systems including FBU, FBM and FBMD are insensitive to the aging process tested in this study.

**Key words:** zirconia, 10 MDP-containing primer, composite resin, artificial aging.

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# Introduction

Nowadays, all-ceramic crowns or bridges are frequently used to provide patients with highly aesthetic tooth coloured restorations. These restorations can be fabricated with a wide variety of materials, such as glass ceramics or zirconia, using many different laboratory processes, e.g. conventional powder modelling or CAD/CAM technology. Although mechanically stable restorations can be fabricated with zirconia cores (1), failures sometimes develop. Beside biological failures, technical failures may include fractures of the complete restoration, chipping or decementation. A 2010 review (2) analysed the stability of all-ceramic FPDs with zirconia frameworks. After five years, 94.2 % of all restorations were still in use, and 76.4 % showed no kind of failure. Chipping was the most common type of failure. Other reviews, published in 2012 and 2013 (3, 4), found 5year fracture rates for all-ceramic tooth-supported FPDs of about 8.1% for molar crowns and about 3.0% for premolar crowns. Core fractures could be seen in 2.5%, and veneer fractures in 3.0% of crowns. Miyazaki et al. (5) concluded that zirconia-based FPDs are promising for dental restorations.

Every dental material is influenced by aging effects during the wearing period in the patient's mouth. In particular, all-ceramic fixed partial dentures (FPDs) like crowns or bridges, as well as onlays or veneers, have to be as resistant to these effects as possible. A variety of different aging effects have been reported, due to many different factors, and these may all influence the long term stability of FPDs. Besides, the mobility of the abutment teeth (6), the design of the ceramic components including the framework (7, 8) and the long-term stability of all-ceramic FPDs may all be influenced by the ceramic material itself (9) or the time of saliva immersion (10). As fracture of allceramic FPDs remains a problem, monolithic fullceramic restorations have attracted increasing interest in recent years. Their advantage is that monolithic restorations can not develop chipping fractures, although fractures of the complete restoration or adhesive failures after cementation can still appear (11).

Cementation is also an important factor for longterm stability; it has been shown that aging processes affect the micromorphological interface between zirconia and resin cement (12). There are various strategies to increase the bond strength between ceramic and adhesive systems. These include modifying the zirconia surface by using mechanical techniques and primer systems (13, 14), or various coating techniques using silica (15), or alumina and aluminium nitride (16).

Moreover, different mechanical and chemical procedures have been described for the intraoral pretreatment of the ceramic surface, in order to improve bond strength to composites (17, 18). For example, Derand showed that grinding with diamond burs could improve bonding between zirconia and adhesive luting cements (19). Modern MDP-containing adhesive systems can bond to an increasing range of zirconia materials, including YPZ (20), TZP (21, 22), YPS zirconia (23) and In-Ceram Zirconia (24, 20). The phosphoric acid groups of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) can react with the oxide layer on the surface of the ceramic materials, which leads to adequate initial adhesion between zirconia and composite (25).

The aim of the current in-vitro study was to analyse the influence of artificial aging through thermocycling and water storage on the shear bond strength between zirconia surface, with two kinds of luting composites for cementation of all-ceramic FPDs and a nanohybride composite for direct restorations, all after surface pretreatment with three modern adhesive systems. This publication is the second part of a study, published in this journal in 2015, entitled "Influence of a light curing 10-MDP adhesive system on the initial shear bond strength of different zirconia composite interfaces" (25).

# **Materials and Methods**

A total of 90 specimens were produced. The same specimen design was used as described in detail in the previous publication (25) (Figure 1).

90 Y-TZP zirconia plates were cut of a pre-sintered Zirconia block (InCoris Maxi-S, Sirona, Germany, Bensheim) using an automatical diamond saw (IsoMet 4000, Buehler GmbH, Germany, Düsseldorf). After that the zirconia plates were sintered in a high temperature oven (LHT 02/17, Nabertherm, Germany, Lilienthal) for 120 minutes at a temperature of 1510 °C following the developer's sintering-instructions. After cooling, the plates were embedded using epoxy resin (EpoThin Epoxy Resin, Buehler GmbH, Germany, Düsseldorf) in round moulds measuring 30 mm in diameter (Ringform 30 mm, Buehler GmbH, Germany, Düsseldorf). The zirconia surfaces were exposed with a rotating diamond disc with 165µm grit, using an automatic polishing machine (PowerPro4000, Buehler GmbH, Germany, Düsseldorf). The specimens were cleaned with alcohol, dried and divided into three groups for three different adhesive surface treatments (FBU = Futurabond U, FBM = Futurabond M+, FBMD = Futurabond M + DCA-Activator). Each of these groups was subdivided into a further three subgroups according to the composite material used (BS = BifixSE, BQ = BifixQM, G = GrandioSO), resulting in nine groups of ten specimens (Figure 2).

A small acrylic glass tube with an internal diameter of 3 mm (Hohlsticks, BEGO, Germany, Bremen) was mounted onto the centre of each ceramic surface with a small portion of sticky wax (Supradent Klebewachs, M+W Dental GmbH, Germany, Bündingen) on the outer side of the tube, but sparing the lumen. All of the groups underwent artificial aging, what is marked with an "A" in the group name.

In groups FBU\_BS\_A, FBU\_BQ\_A and FBU\_G\_A "Futurabond U", a self-etch dual cure universal adhesive (VOCO GmbH, Germany, Cuxhaven) was applied to the ceramic surface. In groups FBM BS A, FBM\_BQ\_A and FBM\_G\_A, a new self-etch, light universal adhesive "Futurabond M+", curing, containing 10-MPD (VOCO GmbH, Germany, Cuxhaven) was applied to the ceramic surfaces. In groups FBMD\_BS\_A, FBMD\_BQ\_A and FBMD\_G\_A, this new adhesive system was mixed with a special dual curing activator (DCA Activator, VOCO GmbH, Germany, Cuxhaven) at a ratio of 1:1. All of the adhesive systems were applied in accordance with the manufacturer's instructions. After the induction period, the overrun of the adhesive fluids was blown away using compressed air, in accordance with clinical practice.

In the corresponding groups, the luting composites Bifix SE or Bifix QM (VOCO GmbH, Germany, Cuxhaven) were applied into the acrylic tube using the manufacturer's application system. The adhesive system and the luting composite were simultaneously light cured. This procedure mimics the clinical practice, e.g. during the adhesive luting of the FPDs. Polymerisation was performed using a polywave-LED polymerisation lamp (Bluephase, IvoclarVivadent, Germany, Ellwangen) for 40 s at 1,200 mW/cm<sup>2</sup>.

In groups FBU\_G\_A, FBM\_G\_A and FBMD\_G\_A, the adhesive system was light cured before application of the composite because the residual tube lumen was filled with a nanohybride material for direct restorations (GrandioSO, VOCO GmbH, Germany, Cuxhaven). After polymerization of the adhesive system, the composite-material was applied into the tube lumen and light cured. This is in accordance with clinical practice during direct composite restorations. Polymerization was performed as described above.

After polymerization of the different composite materials, the sticky wax was carefully removed using a scalpel. Just as the previous publication (25), after 24 hrs initial-storage in distilled water, all specimens underwent artificial aging by thermocycling between 5° C and 55 °C for 5000 cycles and by storage in water at 36°C for 100 days before testing the shear bond strength. Shear bond tests were performed with a universal testing machine (UTS 20K, UTS Test system GmbH & Co KG, Germany, Ulm) 24 hrs after the end of water storage period. Load transfer to the specimens was accomplished with a steel blade with 0.5 mm radius of curvature at its loading edge. The specimens were fixed in a custom designed jig, in such a way that

the blade edge was parallel to the ceramic composite interface and met the composite-containing tube at a distance of 50  $\mu$ m to the interface (figure 3). The test was performed with a cross-head speed of 1 mm/min until fracture occurred (Phoenix – Version V 5.04.006, UTS – Testsysteme GmbH & Co KG, Germany, Ulm). This event was defined by a decrease in load of 5 N. Force at fracture was determined and divided by the ceramic-composite interface area, for conversion into apparent shear bond strength (SBS).

After the fracture, the zirconia-surfaces were evaluated using fluorescence microscopy to identify three possible types of failure which theoretically could have occurred: 1)– adhesive failure between zirconia and adhesive system: In this situation no remaining fluorescent area on the zirconia surface can be found. 2)– adhesive failure between composite and adhesive system: In this case remaining fluorescent areas on the zirconia surface can be found. 3) Cohesive failure within the composite: In this case, fracture of the composite itself can be seen.

Statistical analysis to identify significant influences of the adhesive systems or of the composite materials on SBS was performed by two way ANOVA and the Tukey test. To identify influences of the aging process, the data of the previous publication (25), where SBS without artificial aging was tested, were incorporated into the calculation. Statistical Analysis was performed with "IBM SPSS Statistics V22.0.0.0, 2013, IBM Corp, USA, New York", with the level of significance set to 0.05.



Figure 1. Schematic drawing of the specimen design.



Figure 2. Flowchart of the fabrication of the study groups.



Figure 3. Photograph of the fixation jig (B) and the positioned blade edge (A) before Shear-bond tests

## Results

The measurements of the shear bond tests after artificial aging are shown in table 1. The boxplot in figure 4 compares the data of the corresponding groups without aging (25).

Comparison of the groups FBMD\_BS and FBMD\_BS\_A showed that the aging process had significantly weakened shear bond strength between these groups (p<0.001). No significant effect of aging could be found in the remaining groups.

Furthermore, the data was analyzed with respect to the influence on the shear bond strength after artificial aging of the type of the adhesive system, as well as the type of composite. For both cases, two way ANOVA (table 4) showed significant differences (p<0.001). To identify these differences, Tukey tests were performed. It could be shown that the type of the adhesive system had a significant influence on the shear bond strength of the tested combinations. All of the pairwise comparisons were statistically significant (table 2). As regards the type of composite, it could be shown that the groups with BifixSE as composite material showed significantly lower SBS than Bifix QM (p=0.011) and GrandioSO (p<0.001). There was no significant difference between Bifix QM and GrandioSO (p=0.054) (table 3).

In total, the greatest shear bond strengths between zirconia and either type of adhesive systems after artificial aging were observed in combination with Bifix QM or GrandioSO. Single comparisons between test series BQ and G were not statistically significant. During the shear bond tests, all specimens showed adhesive failures between zirconia and the adhesive system. There were no adhesive failures between the adhesive system and the composite, and also no cohesive failures within the composite, or failures during the aging process.



**Figure 4.** Boxplot showing SBS with the corresponding groups without (25) and after the aging process (marked with an "A" in the group name) plotted next to each other (e.g. FBU\_BS vs. FBU\_BS\_A)

Table 1.Shear bond strength of specimens with different types of adhesive systems (FBU =Futurabond U, FBM = Futurabond M+ and FBMD = Futurabond M + DC-Activator) and differentcomposite materials (BS=Bifix SE; BQ=Bifix QM; G=GrandioSO) before and after artificial aging.Mean, standard deviation, minimum and maximum of SBS are given (sample size n=10).

		FBU		FBM			FBMD			
	Adhesive System	Futurabond U		Futurabond M+			Futurabond M+ DCA			
	Composite	BS	BQ	G	BS	BQ	G	BS	BQ	G
Without artificial aging	Mean [MPa]	10.7	11.2	10.5	19.4	16.1	19.1	17.3	16.6	17.8
	Stand. Dev. [MPa]	5.5	4.6	6.0	5.7	2.5	4.4	5.4	3.4	6.3
	Minimum [MPa]	4.6	6.3	3.9	13.8	13.9	13.1	10.8	11.3	8.7
	Maximum [MPa]	18.9	19.5	18.8	32.2	21.9	26.2	24.1	21.2	27.3
	Mean [MPa]	6.2	12.7	17.4	11.6	15.7	19.2	4.8	14.9	17.3
After artificial aging	Stand. Dev. [MPa]	2.9	5.8	1.9	5.1	8.3	3.3	2.8	5.1	5.4
	Minimum [MPa]	2.6	6.7	15.5	4.6	7.48	13.7	1.0	6.9	7.7
	Maximum [MPa]	11.7	21.9	19.6	18.4	26.6	23.0	10.2	21.6	22.9

Table 2. p values of the single comparison (Tukey-Test) between the adhesive systems.

	Futurabond U	Futurabond M+	Futurabond M+DCA
Futurabond U		< 0.001	0.004
Futurabond M+	< 0.001		0.046
Futurabond M+DCA	0.004	0.046	

 Table 3. p values of the single comparison (Tukey-Test) between the composite systems.

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	Bifix SE	Bifix QM	GrandioSO
Bifix SE		0.011	< 0.001
Bifix QM	0.011		0.054
GrandioSO	< 0.001	0.054	

**Table 4.** Results of the Two way ANOVA, SBS = Shear bond strength.

Source		Type III	Df	Sum of squares	F	p-value
adhesive	SBS	807.042	2	403.521	15 421	< 0.001
system	group	4166.135	2	2083.067	13.421	
composite	SBS	667.653	2	333.827	10 757	<0.001
	group	444.979	2	222.489	12.737	<0.001

# Discussion

In order to generate data, which are comparable to existing data from a previous publication (25), the same sample design was used. Embedded flat zirconia specimens were produced; these were ground automatically with 165  $\mu$ m grit to expose the zirconia surface and to simulate surface roughness after CAD/CAM milling processes. The flat ceramic surface design guaranteed that the samples were reproducible. In clinical work, exposed zirconia surfaces after chipping or delamination fractures are usually curved, although flat surfaces are occasionally found (25).

Derand et al. reported that mechanical surface pretreatment can increase bond strength between zirconia and luting agents (19). Roughening ceramic surfaces with rotating burs or by sandblasting is an established method to increase the adhesive interconnection between ceramic and composite (18, 21, 26, 27). Denry and Holloway concluded that grinding zirconia increases flexural strength as well as crack resistance. They found surface and subsurface damage and microcraters, which were associated with these effects (28). Barragan et al. found SBS between composite resin and zirconia to be 6.9 to 23.2 MPa after different types of mechanical or chemical surface pretreatment (29). It can be concluded that mechanical surface conditioning by sandblasting or by grinding with diamond instruments, as performed in this study, can increase bonding between zirconia and the modern adhesive systems tested.

The current study showed that pretreatment with the new adhesive system Futurabond M+ and mechanically pretreated zirconia improve shear bond strength after artificial aging, compared to existing adhesives. This study is in line with the findings of other researchers, who showed that special primer systems can improve the bond strength between zirconia and composite (30, 31, 32, 33). Matinlinna et al. measured the bond strength of composite to salinized zirconia as 17.6 MPa (34). Kitayamaet al. found that primers containing phosphoric acid or MDP improved the bond to zirconia (35). Foxton et al. showed that MDP-containing primers could improve bond strength without previous surface treatment (36). Otherwise, it has been reported that special primers had no influence on the bond strength between different luting composites and zirconia (37).

The negative influence of the aging processes on the bond strength between zirconia and composites is a problem, which is often discussed in literature. However, in the current study, the new universal primer system Futurabond M+, either alone or in combination with the DCA-Activator, increased shear bond strength between zirconia and BifixQM or GrandioSO after artificial aging (table 1). In test series of FBM and FBMD, the shear bond strengths were higher than in test series of FBU, even though these results did not achieve statistical significance (table 1, figure 3). As in the previous study (25), only adhesive failures at the interface between zirconia and the adhesives were observed. Adhesive failures during the aging process were not observed.

Possible reasons for the failure of all-ceramic FPDs include loss of primary stability of zirconia caused by phase transformation in the intraoral environment (38, 39), or microleakage caused by either polymerization shrinkage of the composite (40) or by the differential thermal expansion of ceramic and composite (33, 41). Microleakage during the aging processes may be caused by hydrolytic processes at the ceramic-adhesive interface and may be a principle reason for the failure of all-ceramic FPDs (42, 43, 44). In 2008, Akgungoret et al. found that bond strength was reduced by nearly 50% after water storage for 150 days. They used airborne particle abrasion and MDP-containing primer with silica coupling agent to bond resin composite to zirconia (17). The findings of the current study showed that modern MDP-containing primer systems can effectively increase the bond strength to zirconia and the stability of these bonds to hydrolysis compared to older systems. The clinical long-term success of zirconia-composite bond, as tested in the current study, must be the subject of further research.

Within the limitations of this study, the results clearly showed that pretreatment of zirconia with MDP-containing adhesive systems can lead to sufficient adhesion between different types of composite and ceramic surface, even after artificial aging. Futurabond M+ and Futurabond M+DCA, which contain 10-MDP, showed the highest SBS in combination with BifixQM and GrandioSO. These higher bond strengths may originate from better wetting of zirconia surface due to improved adhesion to 10-MDP which simultaneously leads to reduced sensitivity to hydrolysis.

#### Clinical relevance

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The new zirconia composite adhesive systems containing 10-MDP enhance initial bond strength (25) and reduce sensitivity to hydrolysis of the zirconia composite adhesion. An increase in the long term stability of all-ceramic FPDs is therefore a promising possibility.

These adhesive systems could simplify work processes for the cementation of all-ceramic restorations in clinical practice as only one of these systems is needed to achieve a sufficiently strong bond between zirconia and composites.

### Conclusion

Within the limitations of the current study, the following conclusions can be drawn:

1. There was no decrease in shear bond strength between zirconia and composites after artificial aging in combination with all of the tested adhesive systems, except BifixSE in combination with FMBD.

2. The tested adhesive systems containing 10-MDP can reduce the sensitivity to hydrolysis of the bond between composite and zirconia.

3. The bond strength does not depend on the type of the tested adhesive systems.

4. The new selective light- or dual-curing adhesive system Futurabond M+, without or mixed with DCA-activator, simplifies clinical practice, as the time for hardening of the adhesive system, for example luting FPDs, can be extended by leaving out the DCA-activator, which might reduce failure during luting processes.

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