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Influence of 10-MDP Adhesive System on Shear Bond Strength of Zirconia-Composite Interfaces

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Abstract

Introduction: This *in-vitro* study investigated the initial 24h bond strength between different composites and zirconia after application of four different adhesive systems. **Methods:** A total of 120 specimens of zirconia (InCoris, Sirona, Germany, Bernsheim) were ground with a 165 µm grit rotating diamond disc. Thirty specimens were each additionally treated with Cimara Zircon “CZ” (VOCO GmbH, Germany, Cuxhaven), Futurabond U “FBU” (VOCO GmbH), Futurabond M+ “FBM” (VOCO GmbH) or Futurabond M+ in combination with the DCA activator “FBMD” (VOCO GmbH). One of three different types of composites – BifixSE (“BS”), BifixQM (“BQ”) or GrandioSO (“G”) (VOCO GmbH) – was bonded to ten specimens each in every group. Shear bond strength (SBS) was determined in a universal testing machine. Statistical analysis was performed with ANOVA and the Tukey test. **Results:** FBM and FBMD gave higher SBS than CZ and FBU in combination with all tested composites. In comparison to FBU, FBM gave statistically significant increases in SBS with BifixSE (19.4±5.7 MPa) (P<0.013) and with GrandioSO (19.1±4.4 MPa) (P<0.021). None of the other comparisons was statistically significant. **Conclusion:** The new 10-MDP-containing adhesive systems FBM and FBMD increases initial SBS between composites and zirconia in comparison to CZ and FBU.

Key words: Zirconia, 10 MDP-containing primer, composite resin, chipping, cementation.

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Introduction

All-ceramic crowns or bridges are frequently used to provide patients with highly aesthetic tooth coloured restorations. Nowadays, such restorations can be fabricated with a variety of dental materials, such as glass ceramics or zirconia, and using many different laboratory processes, e.g. conventional powder modelling or CAD/CAM technology. Mechanically stable veneered restorations can be fabricated with zirconia cores (1), but sometimes veneering-ceramic fractures develop. *Schley et al*. analysed the literature from 1999 to 2009 for information on 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 (2). Fractures can be provoked by high mechanical forces during chewing or by residual stress in the ceramic, e.g. caused by high sintering temperatures or fast cooling rates after sintering (3). In 2012, *Wang et al.* reviewed 37 studies and found 5-year fracture rates for all-ceramic tooth-supported FPDs of about 8.1% for molar crowns and of about 3.0% for premolar crowns. Core fractures could be seen in 2.5% of all cases. Veneer fractures occurred in 3.0% of cases (4). *Dorri* found similar results in a 2013 review (5). In their review *Miyazaki et al.* concluded that zirconia-based FPDs are promising for dental restorations (6).

As fracture of all-ceramic FPDs remains a problem, monolithic full-ceramic restorations have attracted increasing interest in recent years. Their advantage is that monolithic restorations cannot develop chipping fractures, although adhesive failures after cementation can still appear (7). For molar monolithic restorations, zirconia, especially Y-TZP, can also be used. The clinical long-term stability of dental monolithic full-ceramic restorations can be enhanced by crack prevention, after cementing with composite materials. If an adhesively cemented restoration or a repaired fracture is to be successful, it is most important that the bond between ceramic and composite is adequately strong. Therefore, glass ceramic materials can be pretreated using hydrofluoric acid in combination with adhesive systems to achieve an adequate bond to composites. Because of the lack of a glass phase, this technique does not work with zirconia (8). Many different mechanical and chemical procedures have been described for the intraoral pretreatment of the ceramic surface to improve bond strength to composites (9,10). In recent years, several studies have investigated how to enhance bond strength to zirconia (9,14). For example, *Derand* showed that grinding with diamond burs could improve bonding between zirconia and adhesive luting cements (8). Furthermore, modern adhesive systems have become less and less sensitive to technical errors as the usability of these systems has improved. They are able to bond to a wide variety of dental materials: Modern MDP-containing adhesive systems can bond to an increasing range of zirconia materials, including YPZ (17), TZP (13,14), YPS zirconia (15) and In-Ceram Zirconia (16,17). The phosphoric-acid groups of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) can react with the oxide layer on the surface of the ceramic material. This leads to sufficient adhesion between these two materials. These modern MDP-systems are of increasing interest for dental practice, as it will no longer be necessary to keep different types of adhesive systems in stock, for e.g. luting inlays or veneers made of silica ceramics, for repairing veneering fractures or for cementing zirconia restorations (18).

Besides the long-term success of full-ceramic restorations, a high initial stability is important, if e.g. monolithic zirconia onlays are cemented with adhesive systems to change vertical dimension using minimal invasive therapy. The restorations are loaded immediately after cementation and before final correction of occlusal contact points. In this situation a reliable adhesive system and its initial bond strength are important to prevent initial adhesive failures.

Because of this, the aim of the current *in-vitro* study is to analyse the influence of four different modern adhesive systems on the initial shear bond strength between zirconia surface and of two kinds of luting composites for the cementation of all-ceramic FPDs and a nanohybride composite for direct restorations.

Materials and Methods

In total, 120 specimens were made of zirconia. Plates measuring 8.0 mm x 8.0 mm x 2.5 mm were cut out of white-compact Y-TZP zirconia blocks (InCoris Maxi-S, Sirona, Germany, Bensheim) using a diamond saw (IsoMet 4000, Buehler GmbH, Germany, Düsseldorf). Then 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 for the used ceramic.

Zirconia plates were individually 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). To ensure even ablation during the grinding process, zirconia balls (1 mm in diameter) were embedded together with the ceramic plates. After setting, the blocks were ground automatically (PowerPro 4000, Buehler GmbH, Germany, Düsseldorf) with a rotating diamond disc with 165 µm grit to expose the ceramic surface. The specimens were cleaned with alcohol and dried. Then they were divided - first into four groups for four different adhesive surface treatments (CZ = Cimara Zircon, FBU = Futurabond U, FBM = Futurabond M, FBMD = Futurabond M + DC-Activator), and then each of these was subdivided into a further three sub groups according to the composite material (BS = BifixSE, BQ = BifixQM, G = GrandioSO), thus resulting in 12 groups of 10 specimens each (Fig. 1).

In groups CZ-BS, CZ-BQ and CZ-G, the surface was conditioned with a so-called universal ceramic grinding bur (VOCO GmbH, Germany, Cuxhaven) at low pressure and without water cooling, at a rotational speed of 10000 min^{-1}. This surface pretreatment is in accordance with the developers' guidelines for the ceramic repair set Cimara-Zirkon (VOCO GmbH, Germany, Cuxhaven).

Acrylic glass tubes with an internal diameter of 3 mm (Hohlsticks, BEGO, Germany, Bremen) were sectioned into pieces with a length of 3 mm. One of these small tubes was mounted onto the ceramic surface of each specimen 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.

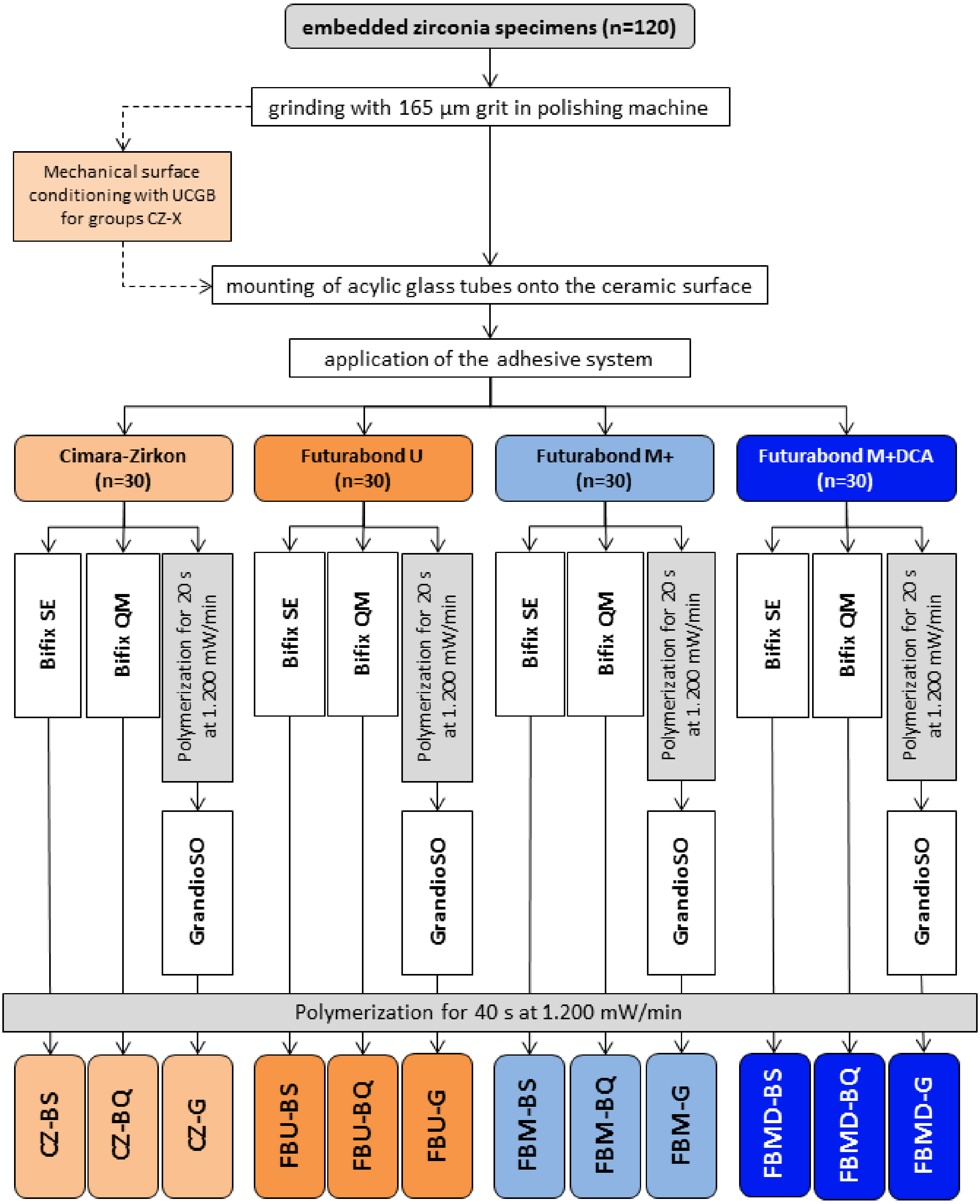

Figure 1. Flowchart of the arrangement of the single study groups. UCGB = Universal Ceramic Grinding Bur (VOCO GmbH, Germany, Cuxhaven).

In Groups CZ-BS, CZ-BQ and CZ-G, an MDP-containing adhesive system (Cimara-Zirkon, VOCO, Germany, Cuxhaven) was applied to the zirconia surface in the tube lumen using a micro brush. In groups FBU-BS, FBU-BQ and FBU-G a self-etch dual cure universal adhesive "Futurabond U" (VOCO GmbH, Germany, Cuxhaven) was used; specimens in groups FBM-BS, FBM-BQ and FBM-G received surface treatment with the new self-etch, light curing, universal adhesive "Futurabond M+", containing 10-MPD (VOCO GmbH, Germany, Cuxhaven). In groups FBMD-BS, FBMD-BQ and FBMD-G, "Futurabond M+" was combined with a special dual curing activator (Futurabond M+ DCA, VOCO GmbH, Germany, Cuxhaven). All of the adhesive systems were applied following the manufacturer's Instructions.

In groups CZ-BS, FBU-BS, FBM-BS and FBMD-BS (Bifix SE, VOCO GmbH, Germany, Cuxhaven) as well as CZ-BQ, FBU-BQ, FBM-BQ and FBMD-BQ (BifixQM, VOCO GmbH, Germany, Cuxhaven), luting composites were used. The composite materials were applied to the acrylic tubes, each using the application system as provided by the manufacturer. In accordance with clinical practice, the adhesive system and the luting composite had to be simultaneously light cured. In groups CZ-G, FBU-G, FBM-G and FBMD-G, 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). Directly after the application of the composite material, each specimen of every group was light cured using a polywave-LED polymerisation lamp (Bluephase, Ivoclar Vivadent, Germany, Ellwangen) for 40 s at 1.200 mW/cm^2. After polymerisation of the different composite materials, the sticky wax was carefully removed using a scalpel.

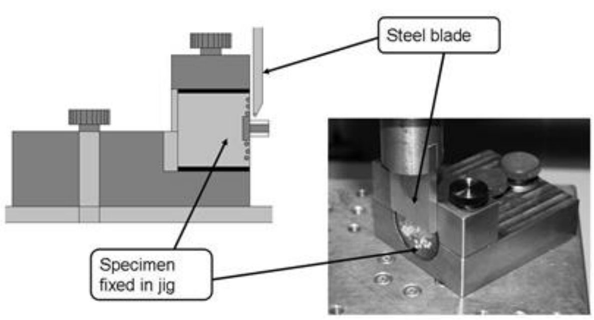

Figure 2. Schematic drawing and photograph of specimen positioned in the universal testing machine.

Shear bond tests were performed with a universal testing machine (UTS 20K, UTS Testsysteme GmbH & Co KG, Germany, Ulm) on the next day. Load transferred to the specimens was accomplished with a bevelled steel blade, mounted to the cross-head of the machine, 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 µm to the interface (Fig 2). 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 removing each specimen from the testing-machine, the ceramic surface was inspected by fluorescence microscopy to evaluate the type of failure. There were three possible types of failure which theoretically could have occurred: 1st – adhesive failure between zirconia and adhesive system, 2nd adhesive failure between composite and adhesive system, 3rd – cohesive failure within the composite. Statistical analysis to identify significant influences of the combination of the adhesive systems with the composite materials on SBS was performed by one-way ANOVA and the Tukey test (IBM SPSS Statistics V22.0.0.0, 2013, IBM Corp, USA, New York), with the level of significance set to 0.05.

Results

The measurements of the shear bond tests are shown in table 1 and fig 3. ANOVA revealed significant differences between the SBS ($P<0.001$). Table 2 shows the p-values of pair-wise comparisons.

In total, the specimens pretreated with Futurabond U (FBU-BS, FBU-BQ and FBU-G) showed statistically significant lower mean bond strength than groups FBM-BS ($P<0.013$) and FBM-G ($P<0.021$), in which the specimens had been pretreated with Futurabond M+. In Group FBMD-G, pretreatment with Futurabond M+ in combination with DCA-activator resulted in significantly higher bond strength than group FBU-BQ ($P=0.039$). No statistically significant difference in bond strength could be found between the remaining groups.

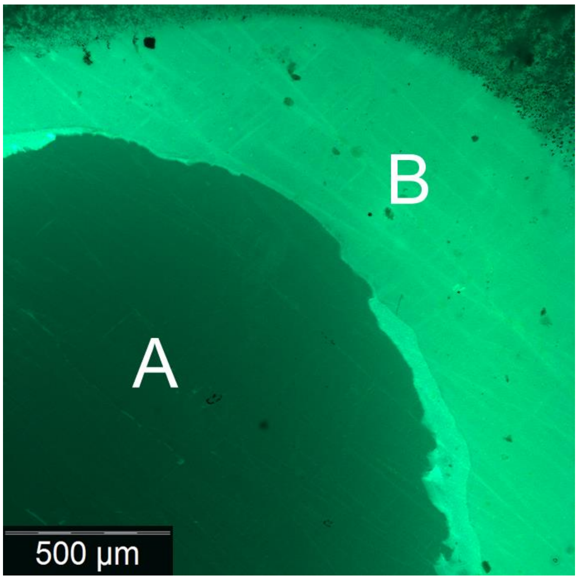

Figure 4. Fluorescence microscopy of a specimen after shear bond test. A = zirconia surface after adhesive failure. B = remaining adhesive in the marginal areas of the zirconia plate, which have had no contact to the composite in the acrylic glass tube.

Single comparisons between groups CZ-BS, CZ-BQ and CZ-G showed no significant difference in shear bond strength between the ceramic surface and the different types of composites after use of Cimara Zircon (P=0.960 and P<1.000). This is similar to single comparisons between groups FBU-BS, FBU-BQ and FBU-G after the use of Futurabond U (P<1.000), between groups FBM-BS, FBM-BQ and FBM-G after use of Futurabond M+ (P<1.000) and between groups FBMD-BS, FBMD-BQ and FBMD-G after use of Futurabond M+ with DCA activator (P<1.000). However, the greatest bond strengths between zirconia and either type of composite were observed after surface pretreatment with Futurabond M+ or with Futurabond M+ with DCA. Single comparisons between test-series FBM and FBMD are not statistically significant. All specimens showed adhesive failures between zirconia and the adhesive system. Adhesive failures between the adhesive system and the composite or cohesive failures within the composite did not occur (Fig. 4).

Discussion

The zirconia plates were cut out of a white compact zirconia block using a diamond saw. This is comparable to the diamond instruments used for milling frameworks or monolithic restorations using CAD/CAM technology. Clinically, flat surfaces can be seen in only a few cases after chipping or delamination, but in most cases the surfaces are curved – especially the luminal surfaces of FPDs. In this study, the zirconia specimens have a flat surface. The flat ceramic surface design of the specimens was chosen, as this guarantees that the samples are reproducible. The zirconia surfaces were ground automatically with a rotating diamond disc with 165 µm grit. This led to surface roughness comparable to that directly after CAD/CAM production and after the use of diamond burs to prepare ceramic surfaces before repair or cementation. In 1977, *Reed* and *Lejus* confirmed that there is an increase in surface hardness up to 4 µm depth because of the t-m phase transformation caused by mechanical surface treatment of zirconia (19). The increase in surface hardness from the t-m transformation is an important factor for the long-term stability of zirconia restorations. *Derand et al.* showed that mechanical surface pretreatment of zirconia increases bond strength to luting agents (8). Roughening ceramic surfaces with rotating burs or by sandblasting is an established method to increase bond strength between ceramic and composite (10,11,13,20). In 2006, *Denry and Holloway* concluded that grinding zirconia increases flexural strength and crack resistance. Furthermore, they found that these effects were associated with surface and subsurface damage and the formation of micro-craters (21).

In clinical situations, it might be necessary to remove luminal interference points using rotating diamond burs before cementing FPDs. This results in surface quality that is nearly equivalent to CAD/CAM production, because the same type of rotating instruments may be used. In 2014, *Barragan et al.* compared shear bond strengths between composite resin and zirconia, with or without sandblasting and chemical surface conditioning with different primer systems. They found mean SBS between 6.9 and 23.2 MPa (22). This study is in the same line to Barragan´s study (Table 1). It can be deduced that mechanical surface conditioning by grinding with diamond instruments – as well as sandblasting – can lead to sufficient bonding between zirconia and the tested modern adhesive systems.

The current study shows that pretreatment with the new adhesive system Futurabond M+ improves shear bond strength between zirconia and composite after mechanical surface treatment with diamond burs. This study is in the same line to the findings of other workers, who showed that special primer systems can improve the bond strength between zirconia and composite (23-26). *Matinlinna* and *Lassila* compared five experimental silanes with a commercially available silane and measured the bond strength to zirconia as 17.6 MPa (27). *Foxton et al.* showed that the use of MDP-containing primers improved bond strength without previous mechanical or laser surface treatment (28). *Kitayama et al.* found that primers containing phosphoric acid or MDP improved the bond to zirconia, while primers containing silane increased bond strength to silica ceramics (29). Otherwise, authors found that special primers had no influence on the bond strength between different luting composites and zirconia (30). In the current study, application of the new self-etch light curing universal primer Futurabond M+ significantly increased the shear bond strength between zirconia and BifixSE or GrandioSO in comparison to the dual cure self-etch universal adhesive Futurabond U (table 2). In test-series FBM and FBMD, the shear bond strength was higher than in test-series CZ and FBU, even though these results did not achieve statistical significance (Table 1,2 and Fig. 2). As expected, only adhesive failures between zirconia and the adhesives were found, because of the high fracture strength of zirconia and the high bond strength between the adhesives and the different composites (Fig. 4).

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

	CZ			FBU			FBM			FBMD		
Adhesive System	Cimara Zirkon			Futurabond U			Futurabond M+			Futurabond M+ DCA		
Composite	BS	BQ	G	BS	BQ	G	BS	BQ	G	BS	BQ	G
Mean [MPa]	13.0	16.0	14.7	8.6	10.4	8.4	19.4	16.1	19.1	17.3	16.6	17.8
Stand. Dev. [MPa]	4.7	3.1	4.5	6.7	5.0	6.9	5.7	2.5	4.4	5.4	3.4	6.3
Minimum [MPa]	7.6	9.3	7.2	4.7	3.2	3.9	13.8	13.9	13.1	10,8	11,3	8.7
Maximum [MPa]	20.0	20.8	21.4	18.9	19.5	18.8	32.2	21.9	26.2	24.1	21,2	27.3

Table 2. Statistical single comparison (Tukey's test) between the groups.

	CZ-BS	CZ-BQ	CZ-G	FBU-BS	FBU-BQ	FBU-G	FBM-BS	FBM-BQ	FBM-G	FBMD-BS	FBMD-BQ	FBMD-G
CZ-BS	-	0.966	1.000	0.997	0.985	0.994	0.139	0.968	0.197	0.732	0.890	0.559
CZ-BQ	0.966	-	1.000	0.485	0.290	0.426	0.912	1.000	0.955	1.000	1.000	1.000
CZ-G	1.000	1.000	-	0.851	0.691	0.806	0.564	1.000	0.671	0.988	0.999	0.956
FBU-BS	0.997	0.485	0.851	-	1.000	1.000	0.013	0.503	0.021	0.190	0.321	0.103
FBU-BQ	0.985	0.290	0.691	1.000	-	1.000	0.003	0.312	0.006	0.087	0.166	0.039
FBU-G	0.994	0.426	0.806	1.000	1.000	-	0.010	0.445	0.016	0.157	0.273	0.083
FBM-BS	0.139	0.912	0.564	0.013	0.003	0.010	-	0.935	1.000	0.999	0.976	1.000
FBM-BQ	0.968	1.000	1.000	0.503	0.312	0.445	0.935	-	0.969	1.000	1.000	1.000
FBM-G	0.197	0.955	0.671	0.021	0.006	0.016	1.000	0.969	-	1.000	0.991	1.000
FBMD-BS	0.732	1.000	0.988	0.190	0.087	0.157	0.999	1.000	1.000	-	1.000	1.000
FBMD-BQ	0.890	1.000	0.999	0.321	0.166	0.273	0.976	1.000	0.991	1.000	-	1.000
FBMD-G	0.559	1.000	0.956	0.103	0.039	0.083	1.000	1.000	1.000	1.000	1.000	-

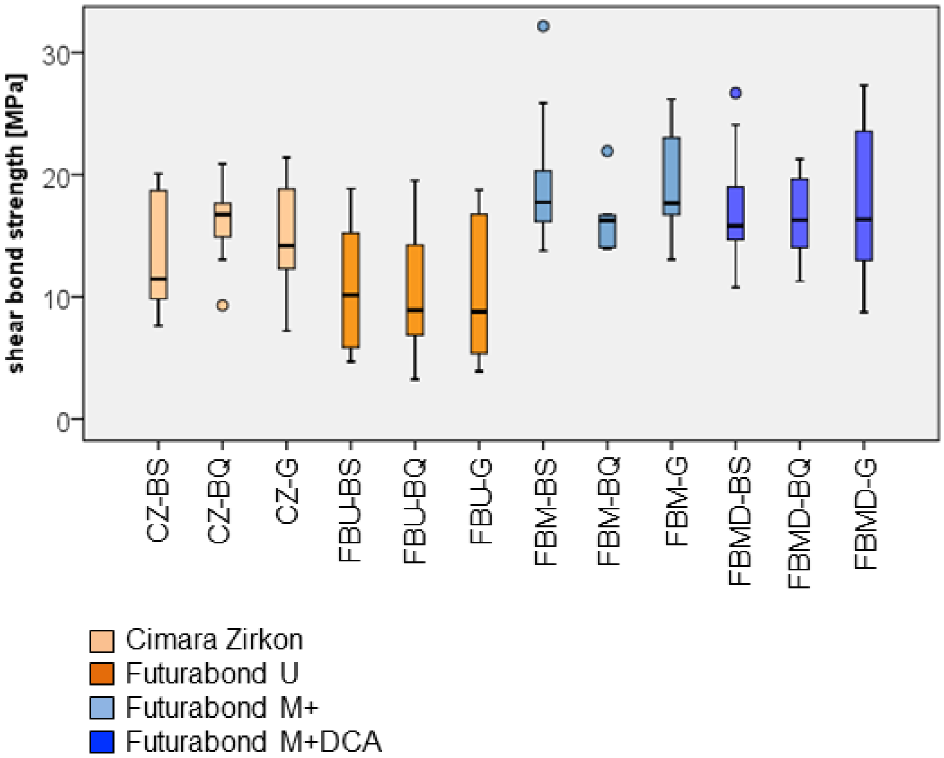

Figure 3. The boxplot shows the median, the upper and lower quartile and spikes of the shear bond strength in the tested groups.

The success of an adhesive bond to zirconia depends on different factors: One possible factor is loss of the primary stability of zirconia by its transformation from the tetragonal into the monoclinic crystallographic phase, as a result of elevated temperature and the presence of moisture (31,32). Furthermore, the material properties of composite may have some influence on the ceramic-composite connection: shrinkage of 1.5 to 3 vol% during polymerisation can cause micro-leakage (33). Similarly, differences in the thermal expansion of ceramics and/or composites may also cause micro-leakage (3,26). Many studies have shown that the stability of ceramic-composite bonding decreases during water storage (9,34,35,36). Micro-leakage between ceramic and composite has been discussed as a possible reason for hydrolysis during long-term water storage. *Akgungor et al*. showed that bond strength was reduced by nearly 50% after water storage for 150 days (9). The influence of artificial aging and the clinical long-term success of the zirconia-composite bonds, as tested in the current study, have to be investigated in further research.

The results of the current study clearly showed that pretreatment of zirconia with MDP-containing adhesive systems leads to sufficient initial adhesion between composite and ceramic surface. Thereby, Futurabond M+ and Futurabond M+DCA, which contain 10-MDP, showed the highest SBS in combination with all of the tested types of composite. These higher bond strengths may originate from better wetting of the zirconia surface due to improved adhesion with 10-MDP.

Clinical relevance

The high initial bond strength is important for final occlusal corrections even some days after cementation without risking a decementaion. In clinical practice, the new system can simplify working with all ceramic restorations, as both the dentist and the dentist's assistant, can use one system for all situations in which a sufficient bond between zirconia and composites is necessary. Furthermore, the light curing material Futurabond M+ simplifies the luting process of all-ceramic restorations. It is possible to check the correct position of the restoration – e.g. of an onlay or a veneer – without being pressed for time while the adhesive system is being chemically cured.

Conclusion

Based on the findings in this study, the following conclusions can be drawn:

Adhesive systems containing 10-MDP can improve the initial bond strength of different types of composite to zirconia.

The initial bond strength does not depend to the type of composite for any of the tested adhesive systems.

The new selective light- or dual-curing adhesive system Futurabond M+ (and DCA) simplifies clinical practice.

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