Effect of surface conditioning methods on the microtensile bond strength of repair composite to indirect restorative materials

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Abstract: This study evaluated the effect of surface conditioning methods on the microtensile bond strength (μTBS) of a restorative composite to indirect restorative materials. Blocks (5 × 5 × 4 mm3) (N = 72) of (a) Zirconia (In-Ceram Zirconia, Vita) (ZR), (b) lithium disilicate glass ceramic (IPS Empress II, Ivoclar Vivadent) (LD), (c) Indirect resin composite (Gradia, GC) (GR) were fabricated (n = 24 per group) and divided randomly into three groups: 1-Control: no conditioning, 2-Silane coupling agent, 3-Hydrofluoric acid (9.5%) (HF)+silane. Each block was duplicated in resin composite. The adhesion surfaces were conditioned with airborne-particle abrasion (110 μm Al2O3 particles). Half of the conditioned blocks received no bonding and the other half one coat of bonding (ED Primer II, Kuraray). Each conditioned block was bonded to a composite block with a resin luting agent (Panavia F2.0, Kuraray). The blocks were sectioned into 1 mm2 microsticks and tested for microtensile bond strength (μTBS) (0.5 mm/min) in a TBS testing machine. Failure types were evaluated under stereomicroscope and Scanning Electron Microscope (SEM). Data were analyzed using three-way ANOVA, Bonferroni corrected and independent sample t-tests ($p < 0.05$). Significant effect of the bonding ($p < 0.001$) and surface conditioning ($p < 0.001$) were observed in all groups. The highest mean bond strength values were obtained in the bonded, HF etched and silanized groups of ZR, LD and GR (12.4 ± 2.9, 28.1 ± 1.5 and 27.2 ± 2 MPa, respectively). HF acid + silane increased the repair bond values in all materials. Majority of the failure types were adhesive for ZR group, whereas HF + silane conditioned LD and GR groups presented predominantly cohesive failures in the cement.

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Keywords: Adhesion; Ceramic; Feldspatic porcelain; Microhybrid composite; Repair; Surface conditioning; Zirconia

Introduction

Aesthetic materials such as glassy matrix or oxide ceramics or resin composites are commonly used for restoring missing teeth or dental tissues. Although a wide range of all-ceramic systems is available on the market, zirconia and lithium disilicate-based ceramics became more frequently accepted in recent years [1]. Chemical composition and mechanical strength of these two ceramic materials differ from each other in that zirconia-based ceramics exhibit twice as much flexural strength than those of lithium disilicate-based ones [1]. Nevertheless, lithium disilicate-based ceramics possess high level of translucency and optical characteristics compared to zirconia due to its high glass content [1].

Despite the increase in the mechanical properties of aesthetic restorations, chipping is one of the most common failure types resulting in replacement of such restorations [2]. In addition to financial reasons, the possibility of trauma to the teeth during restoration removal, difficulty of the removal and the necessity of a long treatment time may delay the replacement of a chipped restoration [3,4]. If the chipped and/or fractured restoration cannot be replaced for such reasons, and the periodontium of the restored teeth is healthy, repair of the restoration should be the first indication [4].

Due to advances in adhesive dentistry, high bond strengths or resin-based composite materials could be achieved that makes the repair of failed restorations an economic and efficient approach. Although the use of zinc-phosphate or resin-modified glass ionomer conventional cements is recommended for luting f zirconia-based restorations [5], such restorations in fact need strong adhesion of the cement for better retention to the teeth due to the precision loss as a consequence of milling in CAD/CAM technologies. Currently, the highest bond strength between ceramic restoration and tooth is achieved using resin cements [6,7]. Additionally, the luting of indirect restorations to the teeth with a resin cement improves the fracture resistance and lifespan of restorations [8].

Numerous surface conditioning methods are suggested in order to enhance the adhesion of resin composites to the restorative materials. However, to date, there is no consensus on the best approach or on the most effective protocol for bonding resin composite to an existing, old-composite restoration for repair purposes [9].

Surface conditioning of restorative materials can be achieved through mechanical roughening [10,11], acid etching [4,12,13], silanization [4,13,14] or a combination any of these methods. For mechanical roughening, the use of aluminum oxide in conjunction with air-borne particle abrasion seems to be a very efficient method [15,16], and it has been well documented that the use of silane coupling agents after air-abrasion increases the chemical bond between the luting resin cement and the restorative material [17]. However, no standard protocol has been demonstrated for repair purposes and the obtained bond strength values vary widely in previous studies [3,16,18]. The repair of ceramic or resin composite indirect restorations with defects is a common treatment option primarily because of its conservative, fast and low-cost characterization compared to fabrication of a new restoration. However, there is limited information on the longevity of repaired resin restorations.

The objectives of this study therefore were to investigate the bond strength of resin composite to indirect restorative materials after different surface conditioning methods for repair purposes and analyse the failure types after debonding. The null hypotheses tested wee that 1) different surface conditioning methods on the indirect restorative materials would not show significant difference in bond strengths and 2) the adhesive resin application would demonstrate no significant effect on the repair bond strength values.

Materials and Methods

Specimen preparation

Blocks (5x5x4 mm^3) (N=72) of zirconia-reinforced alumina-based ceramic (In-Ceram Zirconia, Vita Zahnfabrik Bad Säckingen, Germany (ZR), lithium disilicate-based ceramic (IPS Empress II, Ivoclar Vivadent, Schaan, Liechtenstein) (LD) and photo-polymerizable micro-hybrid resin composite (GC Gradia, GC Corp, Tokyo, Japan) (GR) were fabricated according to each manufacturer`s instructions (n=24 per group). While CAD/CAM zirconia blocks were sintered in a porcelain furnace, lithium disilicate ceramic were prepared according to the pressing program of the manufacturer. Indirect resin composite (GR) was first photo-polymerized and then photo-polymerized under vacuum and heated in an oven (Tescera ATL Processing Unit, Bisco Inc.,

Schaumburg, IL, USA) as per manufacturer`s recommendations. The main materials used in the experiment are listed in Table 1 and representative illustration of experimental design is presented in Fig. 1.

Surface conditioning procedures

The cementation surface of each block was ground-finished using silicon carbide abrasive papers (Buehler, Lake Bluff, IL, USA) in a sequence (600-, 800-, 1000-, 1200-grit) under continuous water irrigation. Each block was cleaned for 10 minutes in an ultrasonic bath (Ultrasonic Cleaner, Biem Ultrasonic Makina San. Ltd., Istanbul, Turkey) in distilled water and air dried. Each block was duplicated in resin composite (Filtek Supreme XT, 3M ESPE, St Paul, MN, USA) using a mould made out of silicon impression material (Elite HD, Zchermack, Badia Polesine, Italy, batch #93058) in order to achieve 4 mm distance between the upper portion of the mould. The resin composite was condensed into the mould by layering, and each layer was photo-polymerized for 40 s using a conventional quartz-tungsten-halogen (QTH) light in standard mode (600 mW/cm_2 output; VIP, Bisco Inc.). Prior to each polymerization cycle, for ensuring the accurate light intensity, the light output was measured with a light meter that was placed on the polymerization unit.

One composite resin block was fabricated for each block. The cementation surface of each ZR, LD and GR block was air-borne particle abraded (AA) with 110 µm grain sized Al_2O_3 particles using an intra-oral air-abrasion device (Micro-Etcher, Danville, San Ramon, CA, USA) at a standardized pressure of 2 bars (0.2 MPa) from a distance of 10 mm for 15 s. The surfaces were then thoroughly cleaned with water spray and air-dried to remove debris. Thereafter, the specimens were randomly divided into three conditioning groups of 12 specimens. In each group, half the conditioned blocks received no adhesive, while in the other half, one layer of adhesive resin (ED Primer II, Kuraray, Tokyo, Japan) was applied according to manufacturer`s instructions.

Group 1 (Control): No conditioning was applied to the bonding surface of the materials. This group was considered as the control group.

Group 2 (Silane): The bonding surface of the materials was coated with a silane coupling agent (Silane Primer, Kerr Corp, Orange, CA, USA, batch #4807393) and allowed to dry for 5 minutes.

Group 3 (HF+Silane): Hydrofluoric acid (HF) (9.5%) (Porcelain Etchant, Bisco Inc, Schaumburg, IL, USA, batch #1400000131) was applied to the surface for 1 minute, rinsed for 30 s, dried with compressed oil-free air for 30 s, and coated with one layer of silane coupling agent as described in Group 2.

For adhesive application, the group without adhesive was named without bonding (-) and the one with adhesive bonding (+). The abbreviations for the subgroups are presented in Table 2.

Bonding procedures and specimen preparation

Each conditioned block was bonded to a resin composite block under a load of 750 g using a dual-polymerized luting cement system (Panavia F2.0, Kuraray, Tokyo, Japan, batch #041113) according to the manufacturer`s instructions. The excess luting cement was removed using a microbrush, and luting cement was photo-polymerized using QTH light (VIP, Bisco Inc) for 40 s from each direction. Oxygen inhibiting gel (Oxyguard II, Kuraray, batch #00631A) was applied on the free surfaces for 10 minutes. After 10 minutes, the bonded blocks were washed and stored in distilled water at 37°C for 7 days before they were tested.

The blocks were fixated on a metal base attached to the sectioning machine. Slices were obtained using a slow-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under copious water cooling. The blocks were positioned as close to perpendicular as possible in relation to the diamond saw. The first section, measuring approximately 0.5 mm was discarded due to the possibility of an excess or absence of cement at the interface that might affect the results. Thereafter, three slices (1±0.1 mm in thickness) were obtained per block. The slices were rotated 90 degrees and once again fixated to the metal base. The first sections were also discarded (1 mm) for the same reasons described above. Two more slices were obtained, each measuring 1±0.1 mm in thickness. This process was followed for the other two groups and only the central specimens were used for the experiments. Six non-trimmed bar specimens with a bonded area measuring approximately 1±0.1 mm^2 and 8 mm length were obtained per block.

Microtensile bond strength test

For microtensile bond strength test **(**μTBS) testing, the obtained beams were fixed with cyanoacrylate glue (Zapit, Dental Ventures of America, Corona, CA, USA) onto a μTBS testing device (Bisco, Schaumburg, IL,

USA), and the specimens were stressed to tensile force loading at a crosshead speed of 0.5 mm/min until failure. After failure, the specimens were carefully removed from the holder, and the adhesive area at the fracture site was measured with a digital caliper (Mitutoyo, Tokyo, Japan). The bond strength was determined by dividing the failure load by the adhesive area, with the result expressed in MPa.

Failure mode analysis

Following μTBS testing, frailure mode of each debonded specimen surface was independently examined using a stereomicroscope (Olympus SZ61, Olympus Co., Tokyo, Japan) at x40 magnification by two calibrated operators. The failure mode was classified as “adhesive” between the specimen and the luting cement, “cohesive” within luting cement, and “mixed” when both types of failures occurred. One representative specimen of each failure mode was examined in a field-emission scanning electron microscope (FE-SEM, Regulus 8230, Hitachi High Technologies Co., Tokyo, Japan) at different magnifications.

Statistical analysis

Data were analyzed using a statistical software package (Number Cruncher Statistical System, 2007 Statistical Software, NCSS LLC, Kaysville, Utah, USA). When evaluating the data, in addition to descriptive statistical methods (mean, standard deviation, median, frequency and ratio), a three-way ANOVA was applied to investigate the multiple effects of group (conditioning methods and bonding variables) on MPa values in comparison of the quantitative findings. For the univariate evaluation of the significance, a one-way ANOVA test was applied, while a Bonferroni Corrected test was used for the control group, and an independent sample *t*-test was used for the other group evaluations. $P<0.05$ was considered to be statistically significant in all tests.

Results

A total of 13 specimens were debonded before the μTBS test, and these groups were included in the statistical analysis with a given value of 0 MPa. The means and standard deviations (SD) of μTBS data for the restorative materials after surface conditioning groups are shown in Table 3.

The results of the 3-way ANOVA revealed that the μTBS values were significantly affected by the material type ($F= 626.390$, $p<0.001$), surface conditioning ($F= 361.949$, $p<0.001$) and bonding application ($F=19.174$, $p<0.001$). Interaction between the material type and surface conditioning factor was significantly different for all treatment groups ($p<0.001$) (Table 4). According to the post-hoc analysis, irrespective of the bonding application and the surface conditioning method, the highest bond strength values were obtained in LD, GR and the lowest for the ZR. The ZR group exhibited the significantly lowest bond-strength values in the control group for both bonding application procedures.

While the highest bond strength values were achieved in bonded LD groups after HF acid and silane application (28.1±1.5 MPa), the control ZR group showed the lowest values (6.99±3.9 MPa). The ranking for μTBS values from lowest to highest were ZR<LD<GR ($p<0.001$) (Table 5). When the silane and HF+silane application were evaluated for both bonding application procedures, the ZR group showed significantly lower results ($p<0.001$) and there was no significant different difference between the LD and GR groups ($p>0.05$). The ranking for μTBS values from lowest to highest were ZR<LD=GR ($p<0.001$) for those groups.

When the effect of the bonding application on the bond strength values of different surface-conditioned indirect restorative materials was evaluated, no significant positive effect was observed ($p>0.05$), except for the HF+Silane treated group ($p<0.001$). HF+Silane treatment in the bonding-applied group demonstrated the highest bond-strength values in all materials and bond strength values were significantly higher than those groups where no bonding was applied ($p<0.001$).

ZR group presented predominantly adhesive failures irrespective of the surface conditioning method (Table 6). For the LD and GR groups, while the control and silane treated groups presented predominantly adhesive failures, the specimens treated with HF+silane showed mostly cohesive failure in the cement. Representative SEM images at different magnifications for each failure type are presented in Figs. 2a-f.

Discussion

In the present study, the effects of different surface conditioning methods and adhesive resin application used during repair procedures were evaluated on the adhesion of resin composite to different indirect restorative materials. Regarding the surface conditioning methods, silane treatment and the HF+silane combination produced positive effects on the bond strength to restorative materials. In addition, the highest bond-strength values were obtained in the LD and GR groups, and values were significantly different from ZR irrespective of the conditioning method applied. Therefore, first null hypothesis could be rejected.

In-Ceram Zirconia tested in the present study is glass infiltrated zirconia with high crystalline content (67% aluminum oxide, 13% zirconium dioxide) and glass phase (20%). Specific conditioning methods are required for resin to adhere to this type of ceramic material [19-22]. Surface conditioning methods such as grinding, abrasion with diamond burs, airborne-particle abrasion (AA) with aluminum oxide, acid etching, silanization or combinations of these methods are often used in repair procedures in order to improve adhesion through roughening and activating the surface [23-26]. AA is the method for surface roughening and cleaning of the restorative materials to improve resin bonding [5,27,28]. However, one of the subjects discussed in the literature on the bond strength of high-strength ceramic materials is the effect of AA. Previous reports propose that mechanical roughening methods such as AA, creates micro-retentions, improving the bond strength between the resins and zirconia, alumina or zirconia reinforced ceramics [7,29,30]. In the previous reports, usually 2.8 bar air-pressure (0.28 MPa) was used for roughening zirconia restoration bonding surfaces [19,20,22,31,32]. However, in the present study air pressure was standardized at 2 bar in order to avoid sub-surface cracks [33,34]. However, the effect of air-abrasion pre-treatment on the tested specimen surfaces was not analyzed in this study.

In addition to AA for roughening the repair surface of the indirect restorations, HF acid (4-9.5%) is generally used as a chemical method for etching [4,7,13,26]. The glassy parts of silica-based ceramics dissolve [12] and a porous, irregular surface [35] with an increased area is produced by HF acid etching. Thus, the resin cement penetrates more easily into the micro-retentions of the etched surface, through which bonding area [5,36] and

wettability are increased [25]. Additionally, silanization promotes wetting of the ceramic surface and thus increases the flow of the resins [37]. In contrast, conventional adhesive cementation methods such as HF acid etching is not effective for glass-infiltrated alumina or zirconia reinforced ceramics [20,21,38-41]. Therefore, various combinations of different mechanical and chemical methods for surface conditioning of zirconia have been suggested to provide a reliable resin bond to zirconia [5]. Various reports on the resin bond strengths for short- and long-term to ZR after different surface conditioning methods [22,40,42]. Indicated that the use of AA with a resin cement revealed high and durable bond strength to zirconia, and the use of a silica coating and resin luting agent combination showed a high, long-term bond strength after water storage and thermal cycling [16,22,39].

In the present study, the highest μTBS value of ZR material was found to be 12.4 (±2.9) MPa in the bonded group of HF acid+silane. Using only silane for pretreatment did not indicate a statistical difference compared to the control group. The reason of these poor bond strengths in ZR might be that the repair surfaces were not coated with silica before silanization. According to these results, surface-conditioning methods used in this study could not enhance bond strength to ZR effectively, as shown in previous reports [30-32,34,36,43]. Moreover, the use of large grain sized (110 μm) particles during AA as well as 2 bar air-pressure might produce lower loss of ceramic material [39] on the bonding surface of zirconia than the other indirect restorative materials tested and hence resulted lower roughness and reduced bond strength. However, after AA procedures, surface roughness analysis on the specimens was not performed in this study. In a previous study [43], the effect of surface conditioning method on the μTBS of a resin cement to high-alumina and zirconia-reinforced ceramics indicated that that silica coating system (30-μm SiO_x) improves bond strength significantly than with grit blasting (110-μm Al_2O_3). Similarly, Amaral *et al*. [22] have also reported that silica coating demonstrated more effective and durable bonding of luting cement to In-Ceram Zirconia ceramic. Therefore, it seems that silica coating system either laboratory or chairside have positive effect on the resin to bond to these ceramic systems but was not used in the present study. Consistently with our findings, the failure modes of the ZR group was predominantly adhesive at the interface between the ceramic and the luting cement, suggesting

that the used surface conditioning method and bonding application had no positive influence on the bond strength values. In addition, most pre-testing failures were also observed in this group.

Using of a phosphate monomer-modified resin cement was recommended to advance a durable bonding to zirconia-based materials for both the short and long-term in several studies [17,22,39,40]. The dual-polymerizing resin cements have been much more preferable for typical use of ceramic restorations due to complete polymerization and great resistance to occlusal loads [44-47]. As in other studies, a dual curing resin containing methacryloyloxy-decyl-dihydrogen-phosphate (MDP) adhesive monomer (Panavia F2.0) was chosen in the present study as an interface material [19,22,27,43,48,49]. In contrast with the outcomes of previous studies reporting durable adhesion of this type of resin luting agent, there were non-tested adhesive failures, especially in the ZR group of the present study. The reason for this could be that there were no efficient surface modifications on the ZR surface, and a lack of silica in the aluminum oxide particles. In addition, it could also be stated that the main indication of the luting agent is adhesive cementation, not repair of ceramic material, as previously reported [50].

Similar to this result, using bonding agents (ED Primer II) that contain MDP did not result in a significant lyhigh bond strength in ZR. The control group of ZR without bonding showed the lowest MPa values (6.9±3.9 MPa). However, the application of adhesive did not show a statistical difference from the non-bonded groups in ZR. Moreover, the application of a bonding agent did not significantly improve the bond strength of tested indirect restorative materials, except for the LD group conditioned with HF and silane in combination. Therefore, the second null hypothesis was also rejected.

In the present study, the highest bond strength was achieved in bonded LD groups with HF acid gel and silane (28.1±1.5 MPa). As reported in a previous study [50], the combination of physical and chemical surface-conditioning methods (HF etching+silanization+adhesive application), as in the present study, was found effective in LD. Bond strength values in bonded LD groups, regardless of surface conditioning methods, were higher than the groups of without bonding. These results show that a bonding agent could be applied with HF acid etching and silanization for a reliable bonding performance in the repair of silica-based ceramics. Likewise,

the failure modes were predominantly cohesive in the cement followed by mixed type of failures in this group suggesting that HF etching+silanization+adhesive application is the optimal surface treatment for the LD ceramic.

Hisamatsu et al. [51] reported that the use of a combined silane and bonding agent showed a remarkable bond strength in repaired indirect composite material. Repair of an indirect restoration with a composite material appears to be an efficient approach due to such advantages as low cost, time saving and the preservation of remaining structures and pulp [52,53]. Hence, the repair of an existing composite restoration with a new composite layer can be more advantageous than creating a new restoration. However, the bond of a new layer composite resin to the existing composite restoration cannot be effective if a suitable surface conditioning method is not used. This is a result of the reactive methacrylate content and the water sorption of the aged composite [54-56]. Different surface treatment protocols such as etching, grinding, and AA followed by adhesive application in various types have been suggested to improve the bond strength of aged and non-aged composites [55,57,58]. For composite repairs, AA with aluminum oxide has shown the greatest bond-strength values by yielding micro-retentive resin composite [24]. In this study, AA with 110 µm Al_2O_3 particles was applied to all repair surfaces to standardize the procedure and contribute by mechanical preparation.

Although it is reported that few significant differences were found in µTBS of resin composites with the use of silane [59], some studies revealed that silanization could increase the bond strength [14,51,60]. As reported previously, silane can create covalent bonds with composite and also increase the wettability of the adhesive that infiltrates into the airborne-particle-abraded micro-retentive surfaces [61]. In addition, acid etching is used before silanization to improve adhesion between resin composites and indirect restorative materials. In the present study, the conditioning of GR material repair surfaces with HF acid+silane and adhesive application revealed higher bond strength values than all of non-conditioned control group and silane groups, and also the non-bonded HF+silane group of GR. Similarly, the failure mode of HF acid+silane and bonding applied specimens in GR exhibited predominantly cohesive in the luting cement, suggesting that this protocol is reliable.

As a result, this study showed that surface conditioning and adhesive application influenced the μTBS of all tested indirect restorative materials. HF acid etching and silanization are the most efficient surface conditioning methods for silica-based ceramics. However, in clinical conditions, chairside silica coating (30 μm SiO_x particles) using an intraoral air abrasion device and silane application could also be considered for improved adhesion of repair resin or luting cement to any of the tested materials in this study due to simpler application procedure rather than a complex HF acid etching, silanization and adhesive resin application [20,43,62,63]. Furthermore, multiple step conditioning protocols could result in operator failures between each stage of the conditioning protocol.

Conclusions

From this study, the following could be concluded:

1- Surface conditioning methods increased repair strength of the resin composite to all indirect restorative materials tested compared to non-conditioned control groups.

2- Regardless of conditioning method, adhesion of resin composite to glass-infiltrated zirconia was inferior compared to glass ceramic and indirect composite with higher incidence of adhesive failures.

3- Hydrofluoric acid etching, silanization and adhesive resin application improved adhesion of resin composite to all indirect restorative materials tested and especially for glassy matrix ceramic.

4- Application of adhesive resin after hydrofluoric acid etching and silanization increased adhesion of resin composite to only glassy matrix ceramic.

Clinical Relevance

Chairside repair with resin composite requires physicochemical adhesion protocol (hydrofluoric acid and silane coupling agent) followed by adhesive resin for glassy matrix ceramic. Application of adhesive resin could be omitted for glass-infiltrated zirconia or microhybrid resin composite.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to figures and tables:

Figures:

Fig. 1. Representative illustration of the experimental design.

Figs. 2a-f. Representative SEM images of failure modes. **a)** Adhesive failure at the interface for the bonding (+) ZR group conditioned with HF+silane at x220), **b)** at (×500). Note the detached bonding or luting cement residues from the surface. **c)** Mixed failure type with very small amount of adhesive component of LD group conditioned with HF+silane at x220, **d)** at x1500. Note the luting cement residues on the surface (arrow). **e)** Cohesive failure in cement of GR group conditioned with silane at x220), **f)** at x1500. Note the remnants of luting cement on the surface (LC, Luting cement).

Tables:

Table 1. The brands, abbreviations, types, shades, batch numbers and manufacturers of the restorative materials used as substrates.

Table 2. Experimental groups, considering the restorative materials to be repaired and surface conditioning methods. See Table 1 for group abbreviations.

Table 3. Means and standard deviations (Mean±SD) of the microtensile bond strength data (MPa) according to the application of adhesive resin. Independent t-test,**p<0.01.

Table 4. Effects of material, surface conditioning and bonding variables on μTBS results (MPa) according to 3-way ANOVA, **p<0.01.

Table 5. Mean±Standard deviations of microtensile bond strength data in each experimental group according to application of adhesive resin (One-way ANOVA with Bonferroni corrected post-hoc analysis).

Table 6. Number of beams analyzed, number of pre-test failures (%) and distribution of failure modes (%) according to tested groups after μTBS test.

Tables:

Material	Material Type	Shade	Batch number	Manufacturer
In-Ceram Zirconia (ZR)	Zirconia		16900	Vita Zahnfabrik, Bad Sackingen, Germany
IPS Empress (LD)	Lithium disilicate glass ceramic	LT B1	V32212	Ivoclar Vivadent, Schaan, Liechtenstein
Gradia (GR)	Indirect resin composite	DA1	160722A	GC Corp, Tokyo, Japan

Table 1. The brands, abbreviations, types, shades, batch numbers and manufacturers of the restorative materials used as substrates.

Restorative materials	**ZR**						**LD**						**GR**					
Surface conditioning	**Control**		**Silane**		**HF acid + Silane**		**Control**		**Silane**		**HF acid + Silane**		**Control**		**Silane**		**HF acid + Silane**	
Adhesive application	–	+	–	+	–	+	–	+	–	+	–	+	–	+	–	+	–	+

Table 2. Experimental groups, considering the restorative materials to be repaired and surface conditioning methods. See Table 1 for group abbreviations.

Material	Surface Conditioning	Adhesive Resin	n	Mean (SD)	Median	Min.	Max.	p
ZR		**Bonding (-)**	24	6.99 (3.90)	7.46	0.00	13.40	**0.999**
	Control	**Bonding (+)**	24	8.35 (3.00)	8.53	0.00	11.90	
		Total	**48**	**7.67 (3.51)**	**8.30**	**0.00**	**13.40**	
		Bonding (-)	24	8.26 (3.38)	8.65	0.00	14.60	**0.999**
	Silane	**Bonding (+)**	24	9.03 (3.76)	8.79	0.00	16.40	
		Total	**48**	**8.64 (3.56)**	**8.68**	**0.00**	**16.40**	
		Bonding (-)	24	11.85 (2.59)	11.08	8.57	16.54	**0.999**
	HF+Silane	**Bonding (+)**	24	12.36 (2.92)	12.07	7.85	16.53	
		Total	**48**	**12.11 (2.74)**	**11.56**	**7.85**	**16.54**	
LD		**Bonding (-)**	24	11.45 (4.25)	12.08	0.00	16.46	**0.999**
	Control	**Bonding (+)**	24	12.78 (3.39)	13.85	0.00	16.44	
		Total	**48**	**12.12 (3.86)**	**12.60**	**0.00**	**16.46**	
		Bonding (-)	24	21.37 (2.65)	22.07	16.58	25.71	**0.999**
	Silane	**Bonding (+)**	24	22.33 (3.39)	23.31	16.18	27.25	
		Total	**48**	**21.85 (3.05)**	**22.66**	**16.18**	**27.25**	
		Bonding (-)	24	23.76 (3.43)	23.80	18.67	28.47	**<0.001****
	HF+Silane	**Bonding (+)**	24	28.12 (1.45)	28.21	25.74	31.48	
		Total	**48**	**25.94 (3.41)**	**26.90**	**18.67**	**31.48**	
GR		**Bonding (-)**	24	15.24 (1.90)	15.29	11.77	18.52	**0.999**
	Control	**Bonding (+)**	24	15.42 (1.71)	15.63	11.92	18.24	
		Total	**48**	**15.33 (1.79)**	**15.56**	**11.77**	**18.52**	
		Bonding (-)	24	21.79 (2.24)	21.58	17.53	25.43	**0.999**
	Silane	**Bonding (+)**	24	22.12 (3.25)	22.28	16.88	27.38	
		Total	**48**	**21.96 (2.77)**	**21.89**	**16.88**	**27.38**	
		Bonding (-)	24	25.30 (4.28)	25.77	17.18	31.30	**0.996**
	HF+Silane	**Bonding (+)**	24	27.20 (2.04)	27.65	22.46	30.62	
		Total	**48**	**26.25 (3.45)**	**26.64**	**17.18**	**31.30**	

Table 3. Means and standard deviations (Mean±SD) of the microtensile bond strength data (MPa) according to the application of adhesive resin. Independent t-test, **p<0.01.

	F	*p*
Model	126.857	<0.001**
Intercept	12907.610	<0.001**
Material	626.390	<0.001**
Surface Conditioning	361.949	<0.001**
Bonding	19.174	<0.001**
Material * Surface Conditioning	36.098	<0.001**
Material * Bonding	2.408	0.091
Surface Conditioning * Bonding	2.653	0.072
Material * Surface Conditioning * Bonding	1.551	0.187

Table 4. Effects of material, surface conditioning and bonding variables on μTBS results (MPa) according to 3-way ANOVA, **p<0.01.

Adhesive Resin	Surface Conditioning Method	Restorative materials			p	Post-hoc
		ZR	LD	GR		
Bonding (-)	Control	6.99±3.90	11.45±4.25	15.24±1.90	<0.001**	ZR<LD<GR
	Silane	8.26±3.38	21.37±2.65	21.79±2.24	<0.001**	ZR<LD, GR
	HF + Silane	11.85±2.59	23.76±3.43	25.30±4.28	<0.001**	ZR<LD, GR
Bonding (+)	Control	8.35±3.00	12.78±3.39	15.42±1.71	<0.001**	ZR<LD<GR
	Silane	9.03±3.76	22.33±3.39	22.12±3.25	<0.001**	ZR<LD, GR
	HF + Silane	12.36±2.92	28.12±1.45	27.20±2.04	<0.001**	ZR<LD, GR

Table 5. Mean±Standard deviations of microtensile bond strength data in each experimental group according to application of adhesive resin (One-way ANOVA with Bonferroni corrected post-hoc analysis).

Material	Surface Conditioning Method	Adhesive Resin	Number of beams	Number of pre-test failures (%)	Distirbution of Failure Modes (%)		
					Adhesive	Cohesive in Cement	Mixed
ZR	Control	(-)	24	4 (16.67)	20 (83.33)	0 (0)	0 (0)
		(+)	24	2 (8.33)	22 (91.67)	0 (0)	0 (0)
	Silane	(-)	24	3 (12.50)	21 (87.50)	0 (0)	0 (0)
		(+)	24	1 (4.17)	23 (95.83)	0 (0)	0 (0)
	HF+Silane	(-)	24	0 (0)	19 (79.17)	5 (20.83)	0 (0)
		(+)	24	0 (0)	18 (75)	6 (25)	0 (0)
LD	Control	(-)	24	2 (8.33)	21 (87.50)	1 (4.17)	0 (0)
		(+)	24	1 (4.17)	18 (75)	5 (20.83)	0 (0)
	Silane	(-)	24	0 (0)	14 (58.33)	8 (33.33)	2 (8.33)
		(+)	24	0 (0)	13 (54.16)	7 (29.17)	4 (16.67)
	HF+Silane	(-)	24	0 (0)	7 (29.17)	11 (45.83)	6 (25)
		(+)	24	0 (0)	6 (25)	10 (41.67)	8 (33.33)
GR	Control	(-)	24	0 (0)	20 (83.33)	3 (12.5)	1 (4.17)
		(+)	24	0 (0)	21 (87.5)	3 (12.5)	0 (0)
	Silane	(-)	24	0 (0)	14 (58.33)	7 (29.17)	3 (12.5)
		(+)	24	0 (0)	12 (50)	9 (37.50)	3 (12.5)
	HF+Silane	(-)	24	0 (0)	9 (37.50)	11 (45.83)	4 (16.67)
		(+)	24	0 (0)	6 (25)	12 (50)	6 (25)

Table 6. Number of beams analyzed, number of pre-test failures (%) and distribution of failure modes (%) according to tested groups after μTBS test.

Figures:

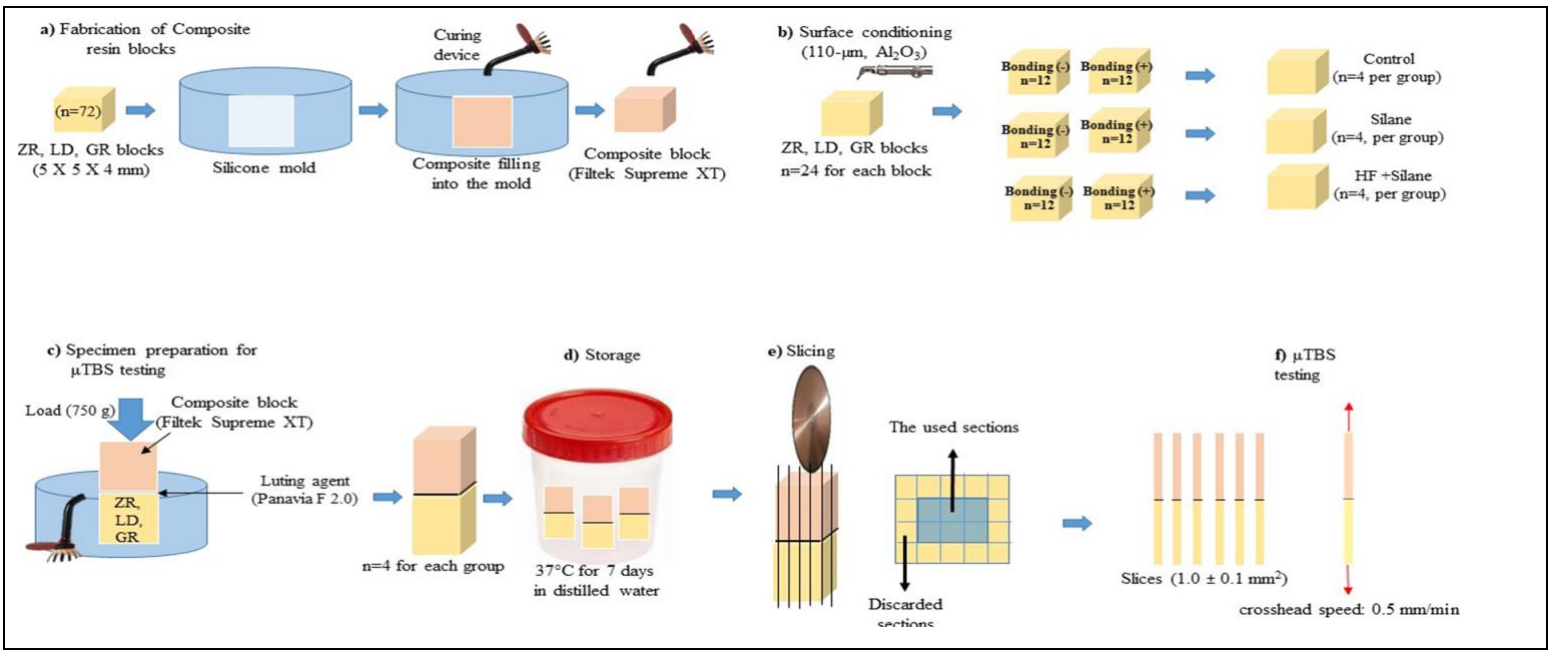
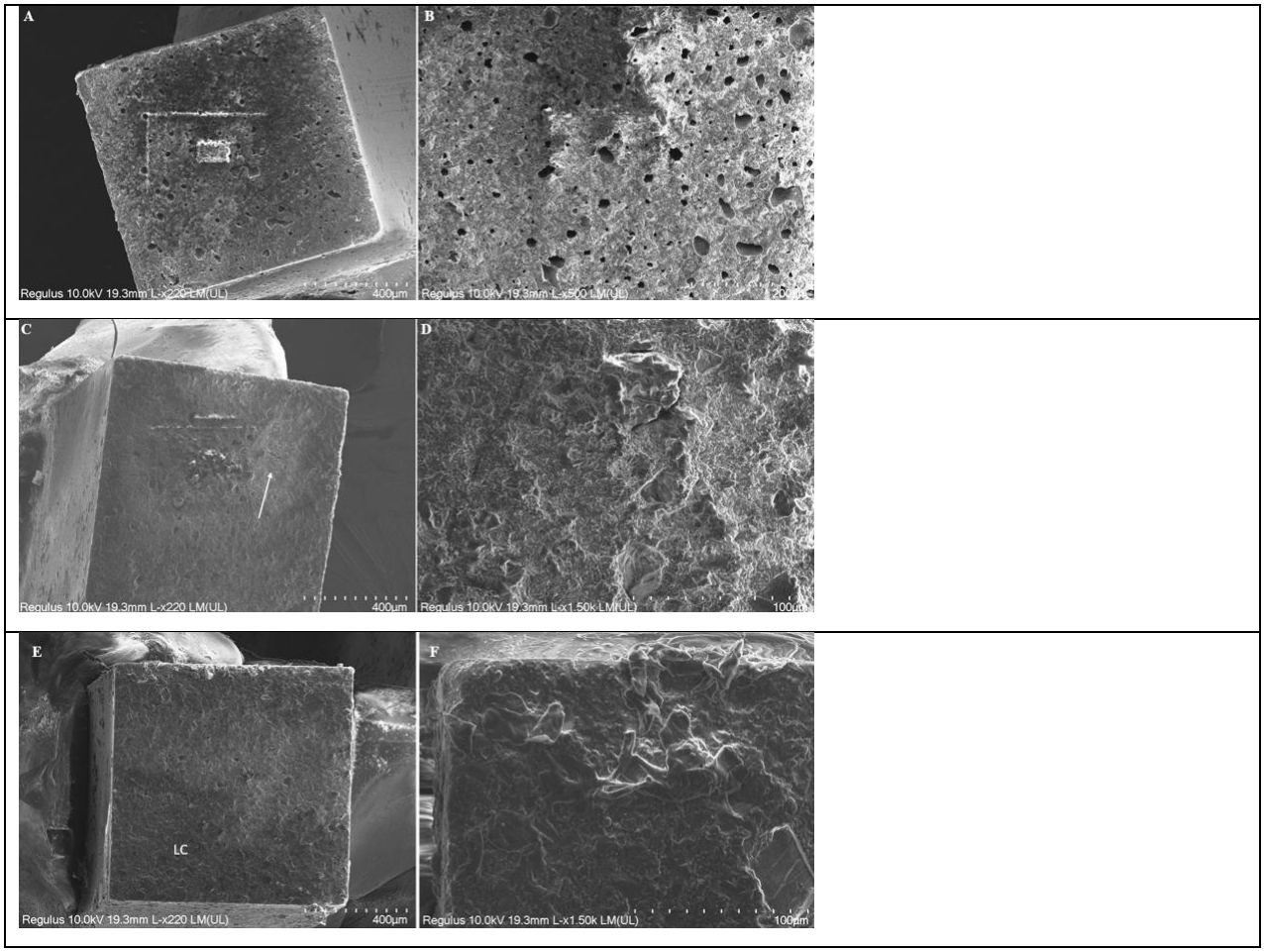


Fig. 1. Representative illustration of the experimental design.



Figs. 2a-f. Representative SEM images of failure modes. **a)** Adhesive failure at the interface for the bonding (+) ZR group conditioned with HF+silane at x220), **b)** at (×500). Note the detached bonding or luting cement residues from the surface. **c)** Mixed failure type with very small amount of adhesive component of LD group conditioned with HF+silane at x220, **d)** at x1500. Note the luting cement residues on the surface (arrow). **e)** Cohesive failure in cement of GR group conditioned with silane at x220), **f)** at x1500. Note the remnants of luting cement on the surface (LC, Luting cement).